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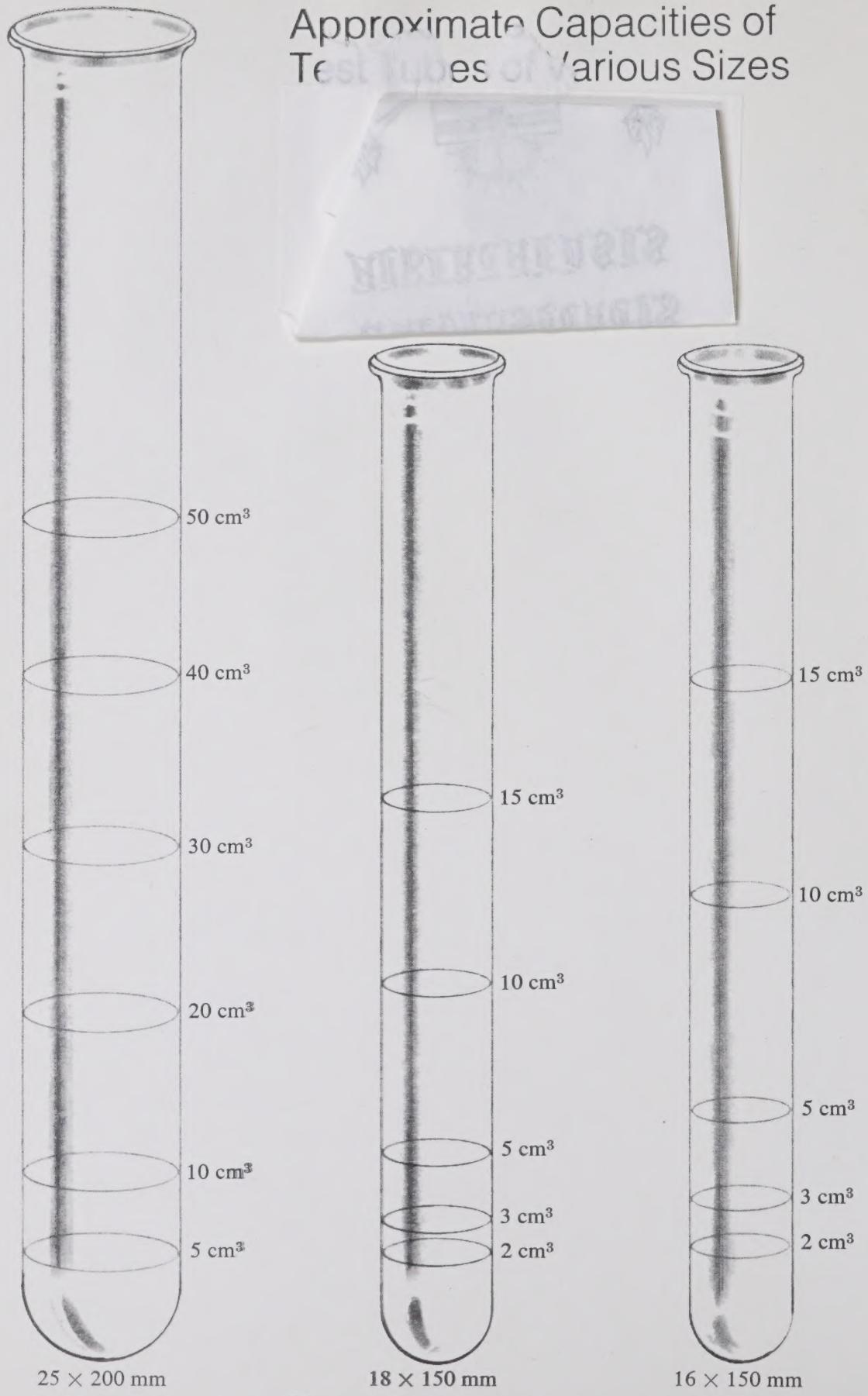
# LABORATORY MANUAL

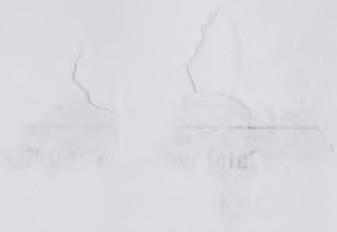
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R. L. WHITMAN  
E. E. ZINCK



# Approximate Capacities of Test Tubes of Various Sizes





# **CHEMISTRY TODAY**

## **LABORATORY MANUAL**

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# INTRODUCTION

Chemists search for answers to chemical problems in the laboratory. This laboratory manual is designed to permit you to do some of the experiments chemists have done and to follow their reasoning as they searched for the answers to their questions.

How much you will gain from the laboratory will depend on you. On one hand, if you simply go through the motions of slavishly following instructions, you will gain very little. On the other hand, if you are interested in what is occurring and why and ask yourself questions regarding every aspect of each experiment, you will profit much from laboratory work.

Numbered questions are posed in the **EXPERIMENTAL** section of each experiment. You should make a conscientious effort to obtain answers to them as you perform the experiment. The main portion of your laboratory report will be made up of answers to questions and supplementary questions along with data tables and mathematical calculations as required.

Ideally, there should be no need for rules concerning house-keeping and safety. However, an ideal system does not exist in chemistry. Certain rules and regulations must be followed in order for you to receive the maximum benefit from your efforts in the laboratory.

## Safety Rules:

1. Never attempt to carry out unauthorized experiments. Your teacher must give approval for all unassigned experiments. *Never work alone* in the laboratory, even during class hours. If you have an accident, there will be no one to help you.
2. Wear safety glasses, regular eyeglasses, or some other form of eye protection at all times in the laboratory. Even though you may feel you are doing nothing dangerous, you have no guarantee that the experiments being performed by your neighbors are not hazardous.

*Note:* Prescription eyeglasses may be purchased with lenses made of safety-glass which is highly resistant to breakage. If shattering does occur the glass breaks into harmless particles instead of sharp slivers.

3. Tie back long hair so that it will not interfere with your work, get caught in the equipment, or catch on fire. Wash your hands occasionally and avoid rubbing your eyes if you have been handling chemicals.
4. Never aim the mouth of a test tube or flask at yourself or anyone else. Never mix concentrated acid solutions with concentrated alkali solutions.
5. *Do not taste chemicals unless directed to do so.*
6. *Be cautious when testing for odors.* Use your hand to fan the odors toward your nose and inhale gently. *Never* hold the material under your nose. If you are directed to use the fume hood, do so. *Never* put your head in the fume hood during an experiment.
7. Be wary of hot glass. Hot glass and cold glass look exactly alike. Teachers become most unhappy when handed a piece of hot glass to examine. Bathe skin burns first in cold water, then in a dilute solution of sodium hydrogen carbonate. If the skin is charred or broken, seek medical help immediately.
8. Learn the location of all fire extinguishers and how to use them.
9. Wash off spilled chemicals on the skin *immediately* with plenty of cold water. This applies especially to the eyes. If there is an emergency face spray or safety shower in the laboratory, learn how to use it. Should someone near you have his eyes accidentally sprayed with a chemical solution he may not be able to open them immediately. Lead him by the arm to the safety flusher and flush his eyes with plenty of water for 5-10 minutes. *Remember:* speed is essential. After all *eye* accidents, the victim should be examined by a doctor.
10. Read the label on a reagent bottle carefully before removing any of its contents. Do not take the reagent bottle to your desk, and do not take any more material than is required. When you have finished with the bottle, return it immediately to its proper place.
11. Ask your teacher to demonstrate how to use a pipet safely. Always see that the tip is kept well under the surface of the liquid you are drawing up into it. For corrosive or poisonous liquids (or liquids which give off poisonous vapors), use a suction bulb instead of your mouth. If you accidentally suck

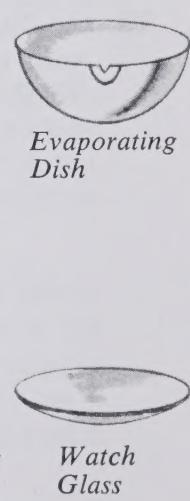
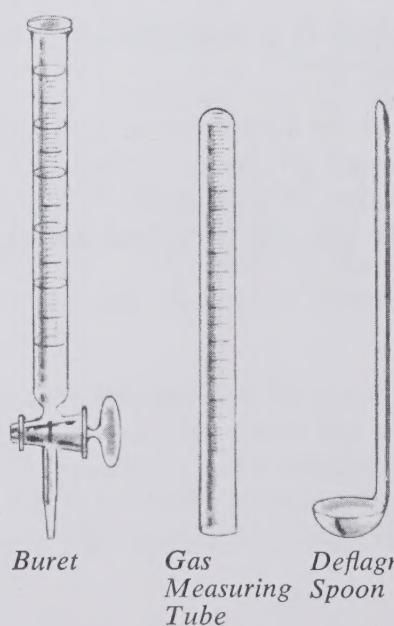
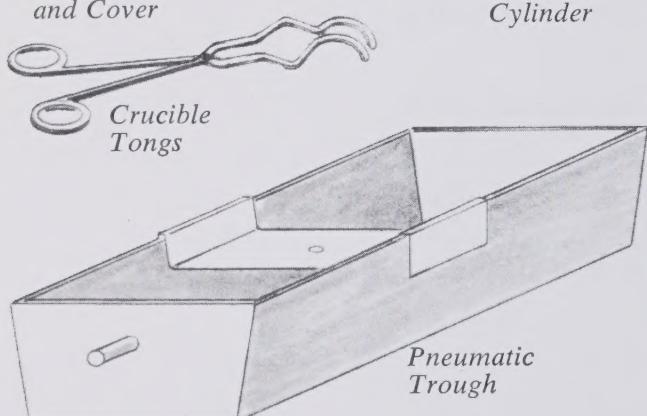
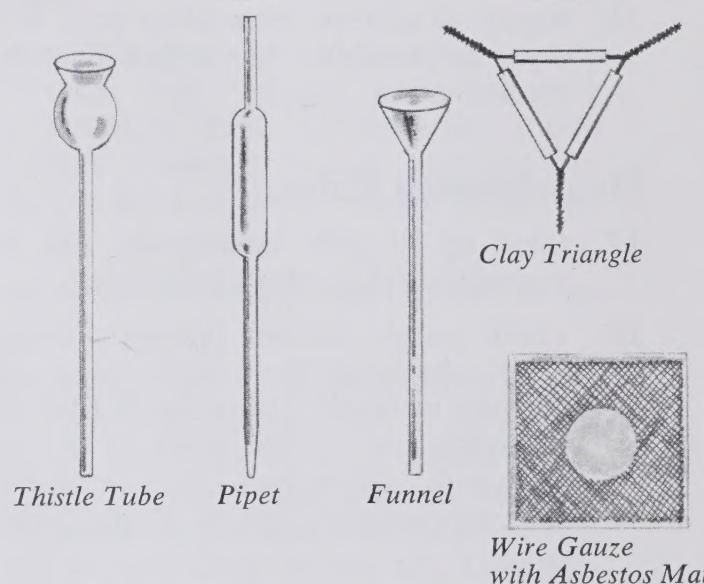
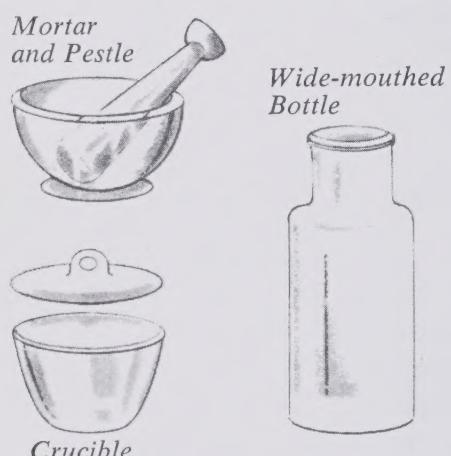
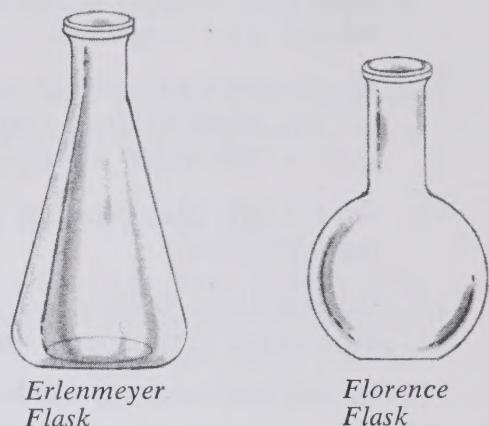
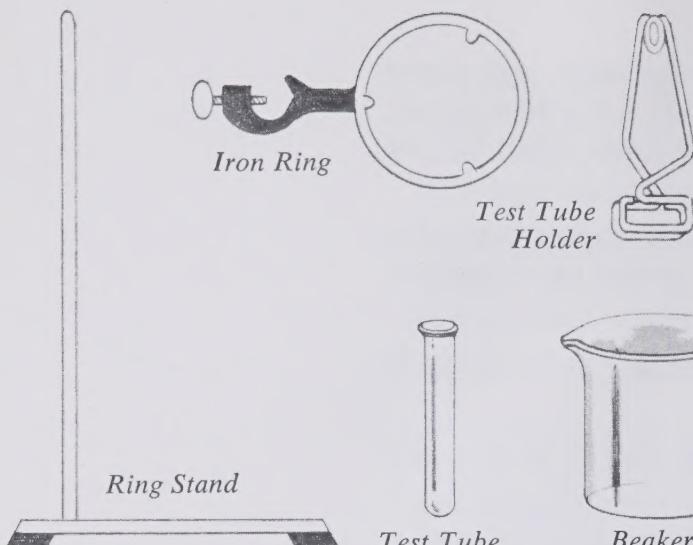
a liquid into your mouth do not swallow it; spit it out *immediately* into the sink (never mind your manners) and wash your mouth out with water 5-6 times. Then tell the teacher what happened.

12. *Never* return any unused material to the reagent bottle. Do not put anything into a reagent bottle except the dropper or spatula with which it is equipped.
13. Do not run, play practical jokes, or engage in horseplay in the laboratory.
14. Do not leave boiling liquids, heated flasks—in fact any experiment in progress—unattended.
15. Do not eat, drink, or smoke in the laboratory.
16. Report all accidents, even minor ones, to your teacher. Also, if you or someone near you feels faint or dizzy, report it to the teacher.

### **Housekeeping Rules:**

17. Clean up all spills immediately. Ask the teacher how to neutralize acid or alkali spills safely.
18. Throw paper, matches (properly extinguished), and insoluble chemicals into a waste jar or wastebasket provided for these materials. *Do not* throw them in the sink. Dispose of liquids and soluble solids in the sink, flushing them down with copious amounts of water. Take special care to pour concentrated acid or alkali slowly into the running water.
19. Be very careful of broken glassware. Ask the teacher where to dispose of it.
20. At the end of the laboratory period, wash and rinse all glassware and return all special equipment to the proper location. Wash and wipe off your desk top and wash your hands. Be sure that the gas and water are turned off.

# Common Equipment Used in This Manual



# INTRODUCTION TO THE LABORATORY

1

## Purpose:

- (a) To become acquainted with the facilities of the chemistry laboratory.
- (b) To make careful observations of a chemical reaction and to attempt to explain the results.

## Introduction:

Your teacher will demonstrate to you the facilities of your chemistry laboratory including the safety facilities and the locations of the various pieces of apparatus which you will be using. If necessary, your teacher will demonstrate the proper use of the laboratory burner and the graduated cylinder.

## Apparatus:

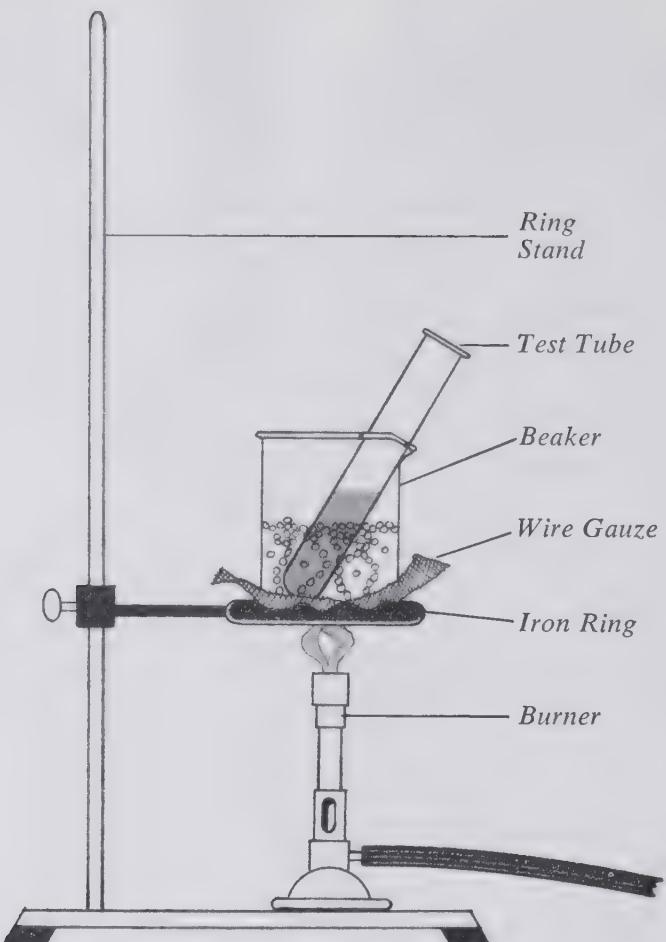
10 cm <sup>3</sup> graduated cylinder	iron ring
150 mm test tubes (4)	ring stand
400 cm <sup>3</sup> beaker	burner
wire gauze	

## Materials:

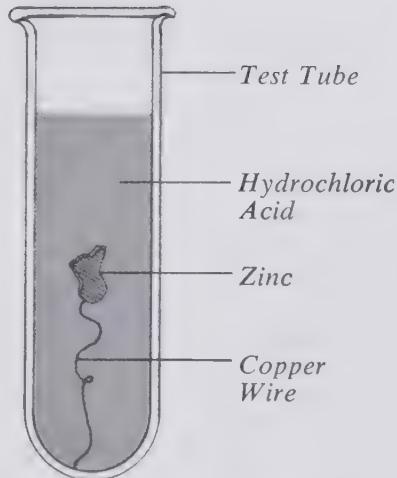
0.25 M hydrochloric acid solution
mossy zinc
copper wire

## Experiment:

- A.** Using a graduated cylinder, place 10 cm<sup>3</sup> of 0.25 M hydrochloric acid in a 150 mm test tube. Add one piece of mossy zinc. (1) *What do you observe?* (2) *If there is a reaction, where is it occurring?* If there is no reaction, warm the test tube in a hot water bath (Fig. 1-1). (3) *If heating is necessary, what is its effect?*
- B.** Using a graduated cylinder, place 10 cm<sup>3</sup> of 0.25 M hydrochloric acid in a second test tube. Add one piece of copper wire. (4) *What do you observe?* (5) *If there is a reaction, where is it occurring?* If there is no reaction, warm the test tube in a hot water bath. (6) *If heating is necessary, what is its effect?*



**Fig. 1-1** Hot Water Bath



**Fig. 1-2** Apparatus for Part C, Experiment 1

- C. Wrap one end of a length of copper wire around a piece of zinc metal as shown in Fig. 1-2. Half-fill a test tube with 0.25 M hydrochloric acid. (7) *What do you predict will happen if the copper-zinc combination is placed in the hydrochloric acid?* Test your prediction. (8) *What do you observe?* (9) *If there is a reaction, where is it occurring?* (10) *Is this result what you would have predicted, knowing the results of parts A and B?*
- D. Try again, using a new piece of zinc wrapped with a new piece of copper wire. Place the metals in a DRY test tube. Add the hydrochloric acid slowly down the inner wall of the test tube until something happens. (11) *What do you observe?* (12) *Are the results obtained in parts C and D similar to each other?* (13) *Are the results of part D what you would have predicted, knowing the results of parts A, B, and C?* (14) *If not, can you propose a reasonable explanation for the results of this experiment?*

# SCIENTIFIC OBSERVATIONS AND MEASUREMENTS

2

## Purpose:

To determine the mass of a given volume of water by different methods and to distinguish between precision and accuracy in the different methods.

## Introduction:

When we attempt scientific measurement, we are concerned with both precision and accuracy. Accuracy refers to how close the measured value is to the accepted or true value. Scientists show how accurate a measurement is by calculating the percent error in the measurement:

$$\text{Percent Error} = \frac{\text{Difference between Accepted Value and Measured Value}}{\text{Accepted Value}} \times 100$$

Precision refers to the number of digits it is possible to obtain in a measurement. A measured mass of 10.32 g is more precise than a measured mass of 10.3 g. (However, neither measurement would be very accurate if the true mass were 10.46 g.) Thus, precision refers to the closeness with which we can make a measurement in a consistent fashion. For example, with a centigram balance we can determine the mass of an object to the nearest centigram (e.g., 10.32 g). Successive, careful measurements of the mass of the same object should agree closely with the first mass obtained. However, with a triple beam balance we can determine the mass of an object only to the nearest decigram (e.g., 10.3 g).

The measurements made on a triple beam balance are never as precise as those made on a centigram balance. However, they would be more accurate if the triple beam balance were properly adjusted and the centigram balance were badly adjusted.

In this experiment you will determine the mass of 1 cm<sup>3</sup> of water using several methods. The accepted mass of one cubic centimetre of water is 1.00 g. You will determine the accuracy and precision of each method you use.

## Apparatus:

100 cm<sup>3</sup> beakers (4)  
100 cm<sup>3</sup> graduated beaker  
100 cm<sup>3</sup> graduated cylinder  
10 cm<sup>3</sup> graduated cylinder

## Experiment:

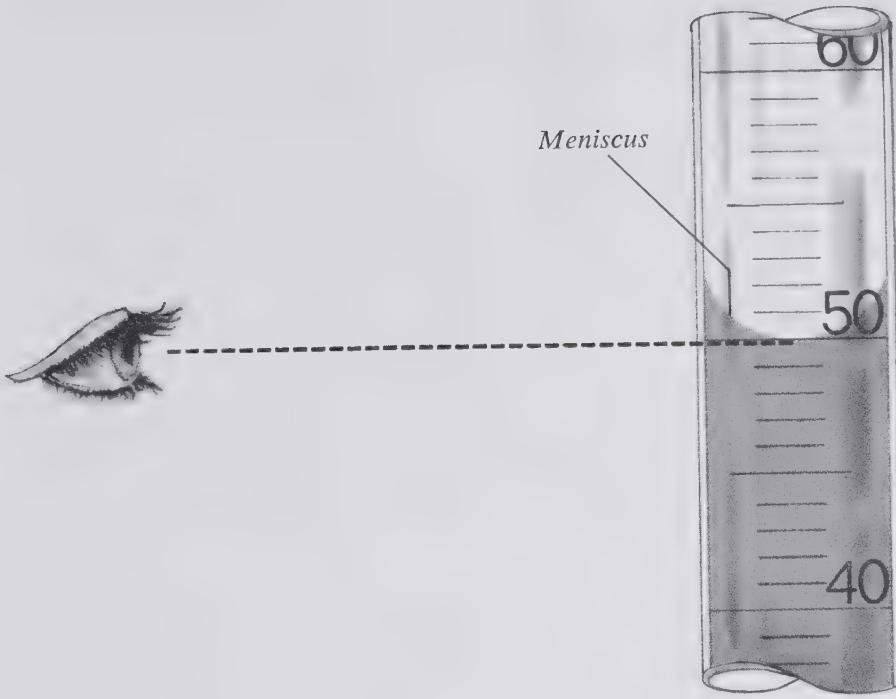
A. Prepare a data table as shown. Record all experimental results as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

DATA TABLE	Part B	Part C	Part D	Part E
Volume of water	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Mass of empty beaker	_____ g	_____ g	_____ g	_____ g
Mass of beaker and water	_____ g	_____ g	_____ g	_____ g
Mass of water	_____ g	_____ g	_____ g	_____ g
Mass of one cubic centimetre of water	_____ g	_____ g	_____ g	_____ g
Percent error	_____ %	_____ %	_____ %	_____ %

B. Determine the mass of a 100 cm<sup>3</sup> beaker. Use a 100 cm<sup>3</sup> graduated cylinder to measure out fifty cubic centimetres of water. Refer to Fig. 2-1 for the proper eye level for reading a volume. Pour the water carefully into the beaker and determine the mass of the beaker and water.

C. Repeat part B using a second 100 cm<sup>3</sup> beaker and a 100 cm<sup>3</sup> graduated beaker to measure out fifty cubic centimetres of water. (1) *In which part (B or C) is the volume determined more precisely? Why?* (2) *In which part (B or C) is the mass of one cubic centimetre of water determined more accurately?*

D. Repeat part B using a third 100 cm<sup>3</sup> beaker and a 100 cm<sup>3</sup> graduated cylinder to measure out ten cubic centimetres of water.



**Fig. 2-1** Proper Eye Level for Reading a Graduated Cylinder

E. Repeat part B using a fourth  $100\text{ cm}^3$  beaker and a  $10\text{ cm}^3$  graduated cylinder to measure out  $10\text{ cm}^3$  of water. (3) *In which part (D or E) is the volume determined more precisely? Why?* (4) *In which part (D or E) is the mass of one cubic centimetre of water determined more accurately?*

### Supplementary Questions:

- (5) *Which part (B, C, D, or E) gives the most accurate measurement of the mass of one cubic centimetre of water?*
- (6) *From your experiment, does it matter whether you use a  $10\text{ cm}^3$  or a  $100\text{ cm}^3$  graduated cylinder to measure out  $10.0\text{ cm}^3$  of liquid?*
- (7) *Do you think it would matter whether you use a  $10\text{ cm}^3$  or a  $100\text{ cm}^3$  graduated cylinder to measure out  $7.3\text{ cm}^3$  of water if accuracy were important? Which cylinder should be used?*

# AN EXERCISE IN OBSERVATION

## Purpose:

To observe results from a series of chemical tests, draw conclusions from these observations, and test the conclusions on an unknown supplied by the teacher.

## Introduction:

In this exercise all your test tubes must be very carefully cleaned and washed with distilled water before use. Dirty apparatus can easily lead you to erroneous conclusions.

## Apparatus:

platinum test wire	cobalt glass squares (2)
test tubes (9)	burner
10 cm <sup>3</sup> graduated cylinder	

## Materials:

6 M hydrochloric acid	0.5 M strontium nitrate
0.5 M sodium nitrate	0.5 M lithium nitrate
0.5 M sodium chloride	0.5 M potassium nitrate
0.5 M barium nitrate	sodium chloride
0.5 M calcium nitrate	

## Experiment:

- Light the burner and adjust it so that the flame is almost colorless. Hold a platinum test wire momentarily in the flame. (1) *What do you observe?* (CAUTION: Do not heat the glass rod into which the platinum wire is sealed. The glass may break or melt if it is heated.) Dip the platinum wire into 6 M hydrochloric acid contained in a test tube and then hold it in the colorless flame of your burner. Repeat the process until the wire no longer imparts a color to the flame. (The wire itself will, of course, become red hot but the flame should not be colored.) (2) *What is the purpose of this procedure?*

**B.** Using a graduated cylinder, measure  $3\text{ cm}^3$  of sodium nitrate solution into a clean test tube. Dip the tip of the platinum wire into the solution and then hold the wire in the flame. (3) *What do you observe?*

**C.** Using the procedure of part A, clean the wire. Using the test tube of sodium nitrate solution for comparison, pour  $3\text{ cm}^3$  of sodium chloride solution into a second clean test tube. Dip the clean wire into the sodium chloride solution and hold the wire in the flame. (4) *What do you observe?*

**D.** Clean the wire again. Dip the hot wire into a little solid sodium chloride and hold the wire in the flame. (5) *What do you observe?* (6) *Is there any difference in the results you obtained in parts B, C, and D?* (7) *What do the results appear to indicate?*

**E.** Using the procedures in parts A and B, test  $3\text{ cm}^3$  samples of solutions of the nitrates of barium, calcium, strontium, lithium, and potassium. Clean the wire carefully after each test. (8) *What do you observe in each case?* (9) *Is there any difference in the results for strontium and lithium?* (10) *If so, what is the difference?* Record all your observations in a data table.

**F.** Again clean the wire and retest your  $3\text{ cm}^3$  sample of sodium nitrate solution, but this time observe the result through two thicknesses of cobalt glass. (11) *What do you observe?* Test the potassium nitrate solution in the same manner. (12) *What do you observe in this case?*

**G.** Now mix the two solutions of sodium nitrate and potassium nitrate. Clean the platinum wire, dip it into the solution, and hold the wire in the flame. (13) *What do you observe?* Repeat, this time observing the result through two thicknesses of cobalt glass. (14) *What do you observe?* (15) *Does this result lead you to any useful conclusion?* (16) *If so, what is the conclusion?*

**H.** Obtain an unknown solution from your teacher and test it in the flame. Observe it also using two thicknesses of cobalt glass. (17) *What can you say about the nature of your unknown?*

## **Supplementary Questions:**

- (18) *Would it have made any difference to your observations if you had used the same cylinder to measure out all the solutions without washing it after each use? Explain your answer.*
- (19) *What do you think is the source of the colors observed in exploding fireworks and during the burning of many brands of artificial logs?*

# METALS AND THEIR CALXES (OXIDES)

4

## Purpose:

To observe what happens when a metal is converted to a calx (oxide) by heating in air and to obtain a metal from its calx.

## Introduction:

The adherents of the phlogiston theory believed that when a metal was heated in air, the metal gave up its phlogiston and became a calx. If the phlogiston were added to the calx, the metal would be regenerated. In this experiment you will observe the formation of two calxes (oxides) and compare the properties of these calxes with the properties of their metals. In the teacher demonstration you will look for a change in mass when a metal is heated. Then you will attempt to obtain copper metal from its calx.

## Apparatus:

crucible tongs	150 mm test tube
burner	buret clamp
evaporating dish	ring stand
spatula	

## Materials:

copper wire
copper oxide
charcoal

## Experiment:

A. *Teacher demonstration.* Support a wire gauze with an asbestos center by resting it on four rubber stoppers (arranged at the corners of the wire gauze) on the pan of a triple beam balance. Place about 5 g of magnesium powder in a small cone in the center of the wire gauze and adjust the sliding masses until the beam is in balance. Ignite the powder with a match or a burner flame. (1) *Is there any evidence of a chemical reaction?* (2) *If so, what is the evidence?* (3) *How does the appearance of the cone at the end of the experi-*

*ment compare with its appearance at the beginning? (4) Is there a change in mass? (5) If so, in which direction is the change?*

**B.** Examine a 15 cm piece of copper wire. (6) *What is its color?* (7) *Does it have a luster?* (8) *Is it easily bent?* Wind the copper wire around a pencil so as to form a tight coil and then remove the pencil. Hold one end of the coil with a pair of tongs and heat the wire strongly in a burner flame until it becomes almost white hot. Do not overheat and melt the copper. Remove the coil from the flame and allow it to cool in the air while still holding it with the tongs. When the coil is cool, examine it. (9) *What is its color?* (10) *Does it have a luster?* (11) *Is it easily bent?*

**C.** To an evaporating dish add equal volumes of copper calx (copper oxide) and powdered charcoal. Mix the two together with a spatula and place the mixture in a test tube. Clamp the test tube to a ring stand and heat the mixture strongly. (12) *Is there any evidence of reaction?* (13) *If so, what is it?* When there is no further change in the mixture, shut off the burner and pour the residue in the test tube back into the evaporating dish. (14) *What does the evaporating dish now contain?* (15) *How do you know?*

### **Supplementary Questions:**

(16) *What word equation would an adherent of the phlogiston theory use to explain the results of part A? Of part B?*

(17) *If you had a sensitive balance, do you think you would find the mass of the copper product after heating to be the same as, greater than, or less than the original mass of the copper? Explain.*

(18) *Based on your observations, what happens to metals when they are heated in air?*

(19) *Why does the filament in an electric light not burn up?*

(20) *What would happen if the glass bulb of an electric light were smashed, leaving the wires intact, and electricity were then passed through the filament?*

(21) *How would an adherent of the phlogiston theory explain the results of part C?*

(22) *How would you explain the results of part C?*

# OXYGEN—ITS PREPARATION AND PROPERTIES

# 5

## Purpose:

To prepare oxygen and investigate some of its properties.

## Introduction:

In this experiment oxygen is prepared by heating potassium chlorate, which causes it to decompose into potassium chloride and oxygen. Manganese dioxide is added to catalyze the reaction so that decomposition will proceed at a lower (therefore safer) temperature than it would in the absence of the catalyst. No oxygen is evolved from the manganese dioxide. All of the manganese dioxide can be recovered unchanged at the end of the experiment.

## Apparatus:

burner	rubber tubing
200 mm test tube	wide-mouthed bottles (3)
buret clamp	glass squares (3)
ring stand	pneumatic trough
one-hole rubber stopper	deflagrating spoon
short glass tube	

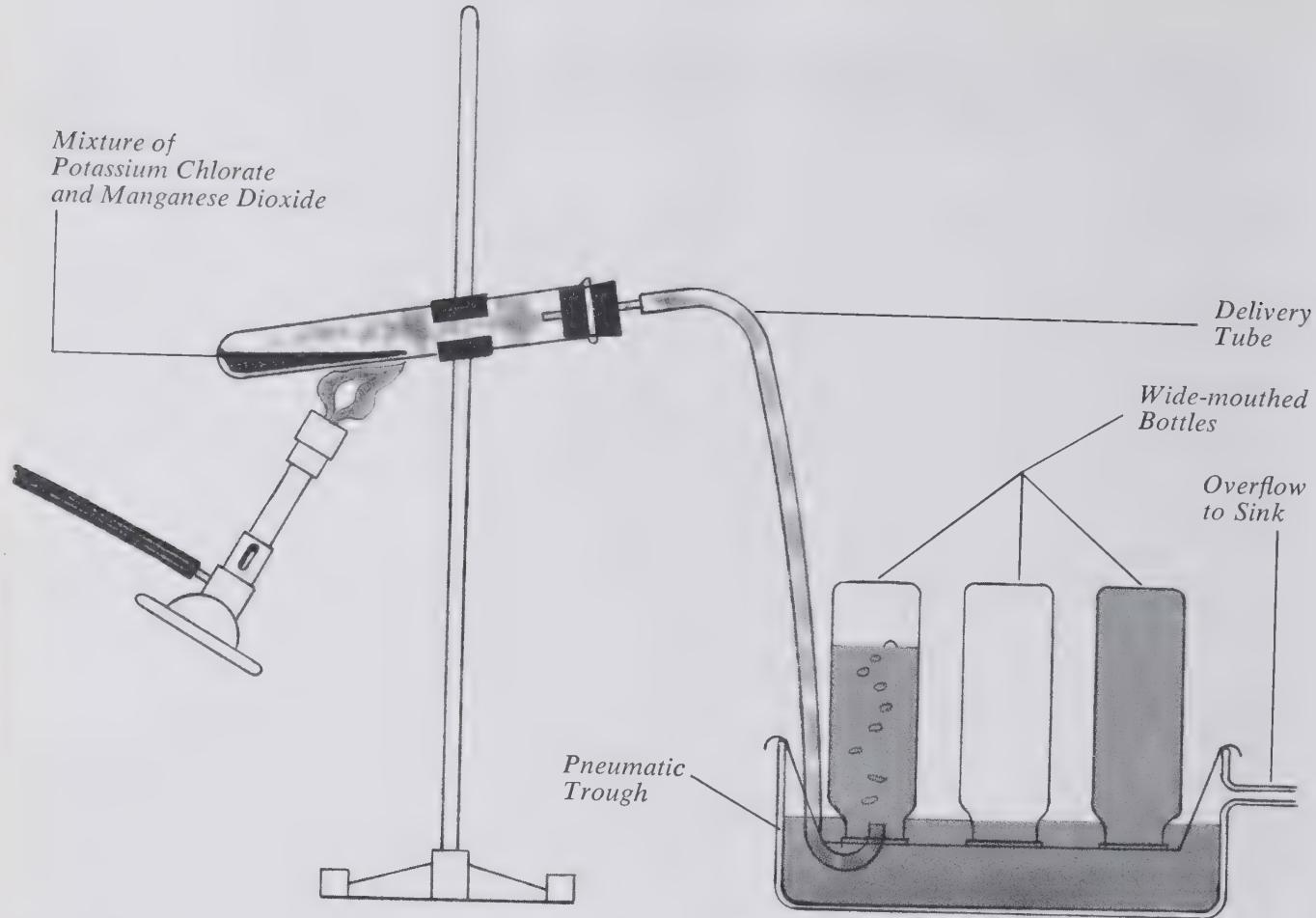
## Materials:

potassium chlorate-manganese dioxide mixture
wooden splint
sulfur
magnesium ribbon

## Experiment:

- A. Using an empty test tube, set up the apparatus for the preparation and collection of oxygen as in Fig. 5-1.
- B. Remove the test tube and place in it 10 g of a mixture of potassium chlorate and manganese dioxide. The mixture has already been prepared, and a test tube containing 10 g of the mixture is on display. Simply fill your test tube to the same level as the one on display.
- C. Spread the mixture evenly over the lower portion of the test tube. Make sure none of the mixture is near the mouth of the tube where the rubber stopper will be inserted.

Mixture of  
Potassium Chlorate  
and Manganese Dioxide



**Fig. 5-1** Apparatus for the Preparation and Collection of Oxygen

- D.** Clamp the test tube in place, making sure the stopper is firmly in position in the mouth of the test tube.
- E.** Fill a wide-mouthed bottle to the brim with water and cover it with a glass square (avoiding air bubbles). Quickly invert the bottle, place it upside down in the trough of water, and remove the glass square. Repeat with two more bottles.
- F.** Insert the delivery tube into the mouth of one of the bottles.
- G.** Heat the potassium chlorate mixture gently and uniformly with a moving hand-held Bunsen burner. Do not heat so strongly that the mixture smokes.
- H.** Fill the three bottles with oxygen. (1) *Is oxygen very soluble in water?*
- I.** Remove the delivery tube from the water, then remove the heat from the test tube. (2) *Why are these two operations not carried out in the reverse order?*

**J.** Place a glass square under the mouth of one of the bottles in the trough and invert it to an upright but still covered position on the desk top.

**K.** Light a wooden splint, then blow the flame out so that the splint remains glowing. Quickly thrust the glowing splint into the bottle of oxygen. (3) *What do you observe?*

**L.** Remove a second bottle of oxygen as in part J. Place a piece of sulphur about the size of a small pea in a deflagrating spoon and light it in your Bunsen burner flame. Quickly lower the deflagrating spoon into the bottle of oxygen. (4) *What do you observe?*

**M.** Remove the third bottle of oxygen. Wind the end of a 5 cm strip of magnesium ribbon around the bottom of a deflagrating spoon. Light the other end of the magnesium ribbon in your Bunsen burner flame. Do not look directly at the burning magnesium; look slightly to one side of it. Burning magnesium emits ultraviolet rays which can be dangerous to the eyes. Quickly lower the burning magnesium into the bottle of oxygen. (5) *What do you observe?*

### **Supplementary Questions:**

(6) *Why must the potassium chlorate mixture be kept away from the mouth of the test tube where the rubber stopper will be inserted?*

(7) *What physical properties of oxygen have you noted during this experiment?*

(8) *Does oxygen burn? How do you know?*

(9) *Does oxygen support combustion? How do you know?*

(10) *At one time astronauts in American spacecraft lived and worked in an atmosphere of pure oxygen. This is no longer the case. Why, do you suppose, was the change made to a different type of atmosphere?*

# PERCENTAGE OF OXYGEN IN AIR

## Purpose :

To determine the percentage of oxygen in the air by volume.

## Introduction :

Lavoisier first determined the percentage of oxygen in the air by heating mercury in air in a closed container. The mercury combined with the oxygen in the air to form mercury calx [mercury(II) oxide]. In this experiment you will use a mixture of pyrogallol and sodium hydroxide to remove the oxygen from an accurately measured sample of air in a stoppered gas measuring tube. When the stopper is removed under water, the water replaces the removed oxygen.

## Apparatus :

battery jar	10 cm <sup>3</sup> graduated cylinder
gas measuring tube	rubber stopper
small test tube	

## Materials :

pyrogallol  
1.0 M sodium hydroxide

## Experiment :

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the table as soon as you have enough data to do so.

DATA TABLE	
Volume of sodium hydroxide required	_____ cm <sup>3</sup>
Volume of gas remaining	_____ cm <sup>3</sup>
Volume of oxygen removed	_____ cm <sup>3</sup>

B. Fill a battery jar with water.

C. Fill a gas measuring tube with water to the 50 cm<sup>3</sup> mark.

- D.** Close the gas measuring tube with a rubber stopper. Turn the tube upside down and measure the volume of air. This represents the volume of sodium hydroxide that is required.
- E.** Pour out the water.
- F.** Measure into a small test tube 0.5 g of pyrogallol (estimated by comparison with 0.5 g contained in a display test tube).  
*(1) Why is it not necessary to measure the pyrogallol accurately?* Add the pyrogallol to the gas measuring tube.
- G.** Measure in a graduated cylinder the required volume of sodium hydroxide solution determined in part D. Pour the sodium hydroxide solution into the gas measuring tube and close it quickly with a rubber stopper. Turn the tube upside down 4 or 5 times to ensure that the contents are well mixed.
- H.** Place the stoppered end of the tube under the water in the battery jar and remove the stopper.  
*(2) Why are these two operations performed in this order rather than in the reverse order?*
- I.** Wait several minutes for the tube to come to the temperature of the room.  
*(3) Why, do you think, is this necessary?*
- J.** Adjust the tube so that the level of water inside the tube is the same as the level of water in the battery jar.  
*(4) Why, do you think, is this necessary?*

### **Supplementary Questions :**

(5) *What is the percentage of oxygen in the air by volume as calculated from your data?*

$$\text{Percent oxygen} = \frac{\text{Volume of oxygen removed}}{\text{Volume of air (50.0 cm}^3\text{)}} \times 100$$

(6) *What is the accepted value for the percentage of oxygen in the air by volume at sea level?*

(7) *How does your result compare with the accepted value for the percentage of oxygen in the air by volume?*

(8) *If the rubber stopper had not been inserted tightly into the gas measuring tube in part G, would there have been any effect on your results? If so, what would have been the effect?*

# 7 MIXTURES

## Purpose:

To prepare a mixture, compare its properties with those of its individual components, and then separate the mixture into its components.

## Introduction:

In this experiment you will study some properties of sodium chromate and of calcium carbonate. Then you will prepare a mixture of the two and compare the mixture's properties with those of its components. Finally you will separate the mixture into its two components by utilizing a difference in one property of the components.

## Apparatus:

150 mm test tubes (4)	ring stand
10 cm <sup>3</sup> graduated cylinder	evaporating dish
mortar and pestle	wire gauze
filter paper	burner
funnel	watch glass
iron ring	

## Materials:

sodium chromate  
calcium carbonate powder

## Experiment:

- A.** In a test tube place a 0.1 g sample of sodium chromate (measured by comparison with a 0.1 g sample that is on display). (1) *What is the color of the sodium chromate?* (2) *What is the form of the sodium chromate?* In a second test tube place a 0.1 g sample of calcium carbonate (measured by comparison). (3) *What is the color of the calcium carbonate?* (4) *What is the form of the calcium carbonate?*
  
- B.** Add 3 cm<sup>3</sup> of water to the test tube of sodium chromate and shake it vigorously. (5) *Is sodium chromate soluble in water?* Now add 3 cm<sup>3</sup> of water to the test tube containing calcium carbonate and shake it vigorously. (6) *Is calcium carbonate soluble in water?*

**C.** Make an intimate mixture of sodium chromate and calcium carbonate by grinding 0.2 g of each together in a mortar. *(7) How does the appearance of the mixture compare with that of the two separate components?*

**D.** Shake the mixture with 10 cm<sup>3</sup> of water in a test tube for 1 min. *(8) What do you observe?* Filter the mixture, collecting the clear liquid filtrate in an evaporating dish. Wash the residue by pouring 5 cm<sup>3</sup> of water down the sides of the filter paper. Place the evaporating dish on a wire gauze supported by an iron ring on a stand and heat gently (to avoid spattering) to evaporate the water. Cover the evaporating dish with a watch glass after most of the water has evaporated, and continue heating gently until all the water has disappeared. Cool the evaporating dish and examine the residue. *(9) What is the residue? (10) How do you know?* Examine the residue on the filter paper. *(11) What is the residue on the filter paper? (12) How do you know? (13) What difference in a physical property did you use to separate the components of the mixture? (14) What are your conclusions about how the properties of a mixture compare with the properties of its components?*

# MELTING POINTS OF SOLIDS

## Purpose:

To determine the melting point of a pure, unknown solid.

## Introduction:

Strictly speaking, the melting point of a pure solid is the temperature at which the solid and the molten liquid can exist together. The melting point is a physical property characteristic of the substance and can be used to identify the substance. In practice, chemists usually do not measure this temperature. Instead, they heat the solid slowly and continuously until it is completely melted. The temperatures between which the first crystal begins to melt and the last crystal disappears define the melting point range of the substance. The slower the rate of heating, the more nearly the melting point range reflects the true melting point of the compound. Impurities in a compound will lower its melting point and cause the melting point range to widen. Pure substances usually have melting point ranges of 1°C or less. Depending on the amount and type of impurity, the melting point range can widen to as much as 20–30°C.

## Apparatus:

watch glass	buret clamp
150 mm test tube	iron ring
melting point tubes	wire gauze
ruler	ring stand
rubber band	burner
thermometer	wire loop stirrer
250 cm <sup>3</sup> beaker	
one-hole rubber stopper for thermometer	

## Materials:

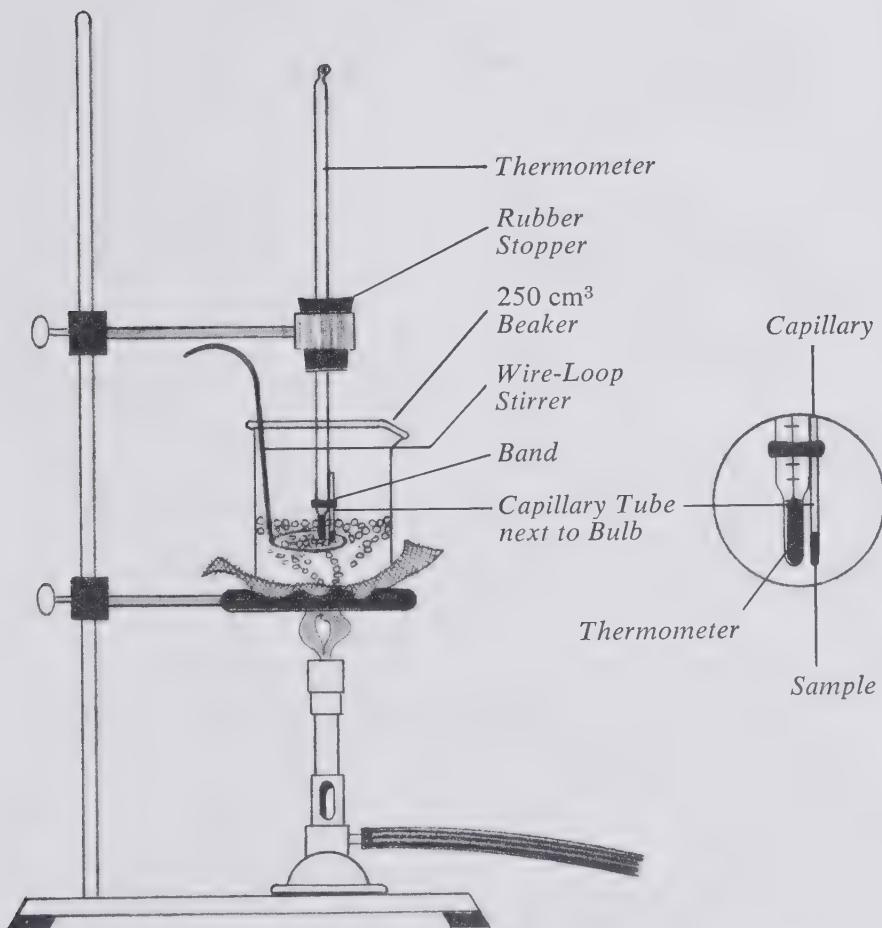
pure, unknown compound

## Experiment:

- A. Place a small quantity of sample on a clean watch glass and pulverize the sample by rubbing it with the bottom of a clean test tube. Do not rub so hard that you break the test

tube or the watch glass. Carefully force a portion of the sample into a melting point tube by pushing the open end of the tube into the powdered sample. Invert the tube and flick it or tap it lightly on the bench to shake the sample into the closed end. Repeat this process until the tube contains sample to a depth of 5 mm. Do not add too much sample to the tube at any one time.

**B.** Place a small rubber band about 5 cm above the thermometer bulb, and insert the melting point tube under the rubber band. The position of the tube should be adjusted so that the sample is located next to the thermometer bulb. Support the thermometer in a  $250 \text{ cm}^3$  beaker about two-thirds full of water. The open end of the melting point tube must be above the water level (Fig. 8-1).



**Fig. 8-1** Apparatus for Determining the Melting Points of Solids

- C.** Heat the water very slowly and with constant stirring. Try to obtain a rate of temperature increase of not more than  $3^{\circ}\text{C}/\text{min}$  ( $1^{\circ}\text{C}/20\text{ s}$ ). Watch the solid in the melting point tube carefully. (1) *At what temperature do the first crystals melt to form a liquid?* (2) *At what temperature does the last crystal just disappear?* (3) *What is the melting point range of your unknown?*
  
- D.** Check the melting point of your unknown with your teacher. If your melting point range is too wide or if your values differ from the accepted value by more than one or two degrees, you will have to make a new determination. Repeat until you obtain acceptable values. Use a new melting point tube for each trial.

### **Supplementary Questions:**

- (4) *Students occasionally find that they can get a  $3^{\circ}\text{C}/\text{min}$  temperature rise, but as they near the melting point their tension increases and temperature rises at a rate of  $5$  or  $6^{\circ}\text{C}/\text{min}$ . What effect, if any, would this have on the melting point range?*
  
- (5) *You are given two colorless solids, each having a melting point range of  $80$ – $82^{\circ}\text{C}$ . Describe a simple procedure by which you would determine whether the two solids are identical or different.*
  
- (6) *What effect does the addition of salt have on the melting point of ice? What practical use is made of this effect?*

# THE BOILING POINT OF A LIQUID

## Purpose:

To determine the boiling point of a pure liquid.

## Introduction:

The boiling point of a liquid is the temperature at which the liquid phase and the gaseous phase of a substance exist together. Like the melting point of a solid, the boiling point of a liquid is a physical property characteristic of a substance and can be used to identify the substance.

## Apparatus:

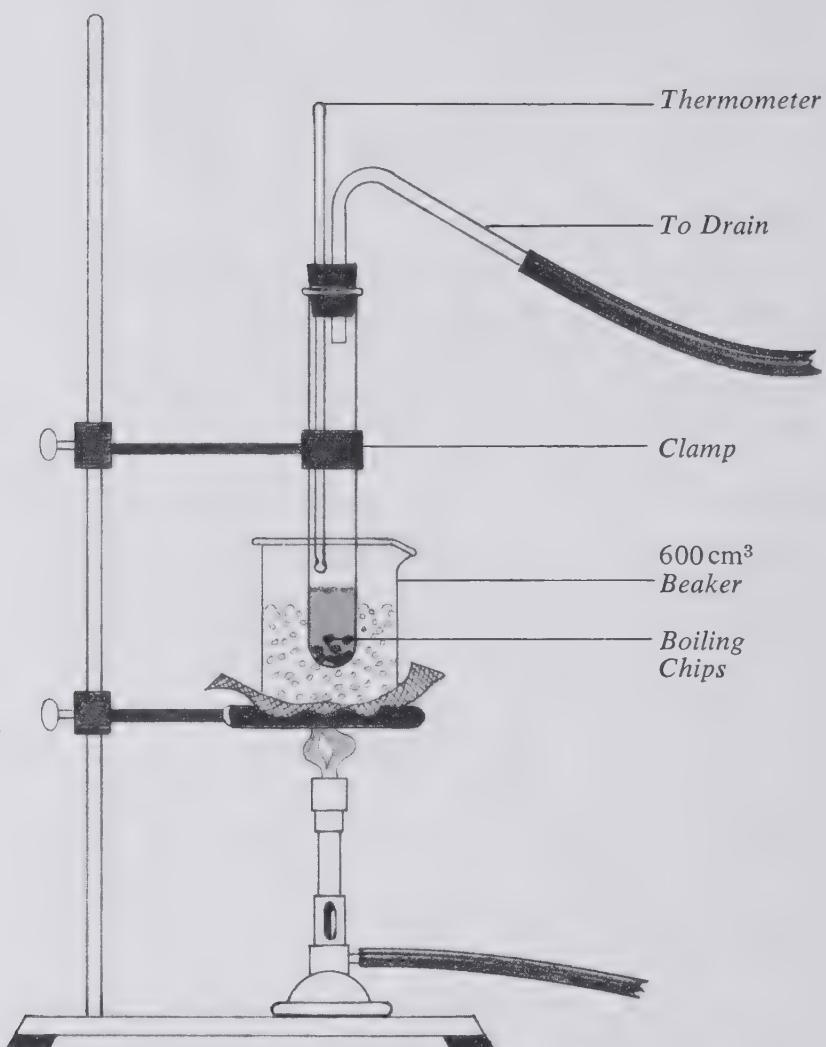
200 mm test tube	wire gauze
two-hole rubber stopper	iron ring
bent glass tube	ring stand
thermometer	buret clamp
rubber tubing	burner
600 cm <sup>3</sup> beaker	boiling chips

## Materials:

pure, unknown liquid

## Experiment:

- A. In a test tube, place a pure liquid sample to a depth of about 3 cm. Place a two-hole rubber stopper fitted with a thermometer and a bent glass tube in the test tube as shown in Fig. 9-1. Add a small boiling chip to the liquid.
- B. The thermometer should be adjusted so that the bulb is about 1 cm above the surface of the liquid. Clamp the test tube to a ring stand and connect a length of rubber tubing to the glass tube. The other end of the rubber tubing is placed in the sink. (1) *What is the purpose of the rubber tubing?*
- C. Support a 600 cm<sup>3</sup> beaker, half-filled with water, on a wire gauze placed on a ring. Immerse the test tube in the water. The test tube should not touch the bottom of the beaker. (2) *Why must the test tube not touch the bottom of the beaker?*



**Fig. 9-1** Apparatus for Determining the Boiling Point of a Liquid

- D.** Heat the water in the beaker gently and watch for changes. (3) *At what temperature does the liquid inside the test tube boil freely? At that point, droplets of liquid should be condensing on the thermometer and falling back down into the boiling liquid in the test tube.* (4) *If you had heated the water bath faster than suggested, what effect (if any) would this have had on the boiling point?* (5) *What is the purpose of the boiling chip?* (6) *How does the boiling chip work?*
- E.** Obtain from your teacher a list of pure liquids and their boiling points. (7) *What is the most likely identity of the liquid assigned to you?*

# PERCENTAGE COMPOSITION

10

## Purpose:

To determine the percentage composition of a sulfide of lead.

## Introduction:

In the early days of chemistry a great controversy arose between two French chemists. J. L. Proust maintained that a compound always had the same composition no matter how it was prepared. His compatriot, C. M. Berthollet, however, argued that a compound should have an infinite number of compositions, depending on the proportions of its components that were used during its preparation. By comparing your results and those of your classmates, you will be able to decide who was correct.

## Apparatus:

crucible and cover	burner
clay triangle	crucible tongs
iron ring	scissors
ring stand	

## Materials:

lead foil
sulfur

## Experiment:

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the table as soon as you have enough data to do so.

DATA TABLE	
Mass of crucible and cover	_____ g
Mass of crucible, cover, and lead	_____ g
Mass of lead used	_____ g
Mass of crucible, cover, and sulfide of lead	_____ g
Mass of sulfide of lead	_____ g
Mass of sulfur in sulfide of lead	_____ g

- B.** Determine the mass of your crucible and cover to the nearest 0.01 g.
- C.** Obtain a piece of lead foil (about 1 g) from your teacher. Using scissors, cut the lead foil into the smallest possible pieces. Place the lead pieces in the crucible and determine the mass of the crucible, cover, and lead. *(1) Is it desirable or necessary that everyone have exactly the same mass of lead foil?*
- D.** Add enough powdered sulfur to cover the lead completely. *(2) Why was the mass of sulfur to be used not specified?* Cover the crucible. Attach an iron ring to a ring stand. Support the covered crucible in a clay triangle on the ring.
- E.** Heat the mixture gently at first, then more vigorously until the excess sulfur begins to burn at the edge of the cover. *(3) What is a good description of the odor of the burning sulfur?* Continue heating strongly until all the excess sulfur has been removed.
- F.** Remove the crucible, allow it to cool for 10 min and obtain the mass of the crucible, cover, and contents.

### **Supplementary Questions:**

- (4) What percentage of the mass of the sulfide of lead is contributed by the lead?*
- (5) What percentage of the mass of the sulfide of lead is contributed by the sulfur?*
- (6) How do your percentages compare with those of your classmates?*
- (7) What might be some of the reasons why an individual result is much higher or lower than the majority of results?*
- (8) Which of the two French chemists, do you think, had the correct hypothesis?*

# DESIGNING A MODEL

## Purpose:

To devise a model to represent an object in a sealed box which can never be opened.

## Introduction:

Often people involved in science are forced to try to visualize objects which they do not expect to see. The atom is a good example. Scientists have reason to believe that they will never actually see the inside of an atom. However, they have been able to construct a model of the structure of the atom based on experimental evidence.

Bohr developed a solar system model of the atom, but further experimentation led to the development of the quantum mechanical model of the atom which we use today.

We can place ourselves in the same kind of situation in which scientists find themselves with respect to the atom. Consider an object of unknown size, shape, density, and composition sealed in a box. The box can never be opened, and we will never be able to see the object. However, we can perform operations on the box which will help us to learn more and more about the object. Finally, we should be able to develop a mental picture or model of the object.

## Apparatus:

box containing an object

## Experiment:

- A.** Obtain a box containing an unknown object. Perform a series of manipulations on the box and observe the results of each manipulation. You may do anything to the box as long as you do not open or damage it. (1) *Why must the box not be opened?* You can shake and tilt the box and obtain its mass. (An empty box of the same size will be provided so that you can obtain its mass as well.) (2) *What manipulations did you perform, and what did these yield?*
- B.** Try to develop a mental picture of the object inside the box. (3) *What is your model of the object?* Test your model by doing further manipulations. (4) *What manipulations did you perform to test your model, and what did these yield?* (5) *Did the results from your manipulations force you to*

*modify your model? (6) What very detailed description of the object in the box can you write?*

### **Supplementary Questions:**

- (7) *What were your most useful manipulations and what made them so useful?*
- (8) *What were your least useful manipulations and why were they not as useful as you had hoped?*
- (9) *What other manipulations would you have liked to perform but for which you did not have the necessary equipment?*
- (10) *What information had you hoped to get from the manipulations you were not able to make?*

# THE VARIATION OF ATOMIC PROPERTIES

12

## Purpose:

To determine whether there is any regular variation in the properties of elements when the elements are arranged in order of increasing atomic number.

## Introduction:

In this exercise you will plot a property (atomic volume, atomic radius, or ionization potential) against the atomic numbers of the elements. You will examine the graph in an attempt to find out whether there are any regular variations in the property.

## Procedure:

- A. Your teacher will tell you which of the properties (atomic volume, atomic radius, or ionization potential) you are to plot. The data are given in Table 12-1.
  
- B. Design the scales for your graph on the graph paper provided. Divide the vertical axis into intervals of convenient size so that the largest numerical value of the property you are plotting will appear near the top of the axis. Label the vertical axis with the name of the property. Divide the horizontal axis into 36 intervals using as much of the axis as possible. Label this axis *atomic number*.
  
- C. Plot the datum for each element. Join consecutive points with solid straight lines. When a datum for an element is missing, join the points by a broken straight line. (1) *Does the portion of the graph for elements 3 to 10 exhibit any similarity to the portion of the graph for elements 11 to 18?* (2) *Does the portion of the graph for elements 19 to 36 exhibit any similarity to the portion of the graph for elements 11 to 18?* (3) *Does the graph illustrate a periodic variation (i.e., regular repetition) between the property you have plotted and the atomic numbers of the elements?* (4) *If there is a periodic variation in the property, describe it.*

TABLE 12-1 ATOMIC PROPERTIES

Atomic Number	Element	Atomic Volume (cm <sup>3</sup> /mol of atoms)	Atomic Radius (nm)	Ionization Potential (V)
1	H	14.0	0.030	13.6
2	He	29.2	0.093	24.6
3	Li	13.0	0.152	5.4
4	Be	4.9	0.111	9.3
5	B	4.4	0.088	8.3
6	C	6.2	0.077	11.3
7	N	14.0	0.070	14.5
8	O	14.3	0.066	13.6
9	F	16.7	0.064	17.4
10	Ne	18.0	0.112	21.6
11	Na	23.6	0.186	5.1
12	Mg	14.1	0.160	7.6
13	Al	10.0	0.143	6.0
14	Si	11.4	0.117	8.1
15	P	17.0	0.110	11.0
16	S	16.4	0.104	10.4
17	Cl	14.2	0.099	13.0
18	Ar	28.4	0.154	15.8
19	K	44.7	0.231	4.4
20	Ca	25.9	0.197	6.1
21	Sc	14.8	0.160	6.6
22	Ti	10.7	0.146	6.8
23	V	8.5	0.131	6.7
24	Cr	7.6	0.125	6.8
25	Mn	....	.....	....
26	Fe	7.1	0.126	7.9
27	Co	6.8	0.125	7.9
28	Ni	6.7	0.124	7.6
29	Cu	7.1	0.128	7.7
30	Zn	9.2	0.133	9.4
31	Ga	11.8	0.122	6.0
32	Ge	13.3	0.122	8.1
33	As	13.0	0.121	10.0
34	Se	18.5	0.117	9.7
35	Br	24.9	0.114	11.8
36	Kr	38.5	0.169	14.0

## Supplementary Questions:

- (5) *What do you estimate to be the missing value of the property for manganese?*
- (6) *Would you expect the value of the property you plotted to be higher or lower for element 37 than for element 36?*

# BOND TYPES AND PHYSICAL PROPERTIES

## Purpose:

To study the effect of bond types on the physical properties of a substance.

## Introduction:

Some substances have ionic bonds. They consist of positive and negative ions arranged in a symmetrical manner. The attractive forces between the oppositely charged ions are responsible for holding the particles together in an ionic crystal.

Other substances consist of molecules with covalent bonds. The atoms in covalent molecules are held together by the sharing of electrons between nuclei.

In this experiment you will examine four physical properties (odor, hardness, melting point, and solubility) of two solids, and you will attempt to relate these properties to the type of bond present in each solid.

## Apparatus:

filter paper	iron ring
watch glass	ring stand
spatula	burner
crucible cover	150 mm test tubes (4)
clay triangle	10 cm <sup>3</sup> graduated cylinder

## Materials:

sodium chloride  
naphthalene  
benzene

## Experiment:

- A. On two pieces of filter paper place separate 0.2 g samples of sodium chloride and naphthalene ( $C_{10}H_8$ ). (1) Is sodium chloride a covalent or an ionic solid? (2) Is naphthalene a covalent or an ionic solid?
  
- B. Note the odor of each solid. (3) What is the odor of sodium chloride? (4) What is the odor of naphthalene? (5) What does

*an odor indicate about the ability of the molecules of a solid to leave the surface of the solid?*

C. Rub a small sample of each solid between your fingers. Note whether each solid feels soft or hard. Check by attempting to crush a few small crystals of each between a watch glass and a spatula. (6) *Are crystals of sodium chloride hard or soft?* (7) *Are crystals of naphthalene hard or soft?*

D. Place a few crystals of sodium chloride and naphthalene side by side on an inverted crucible cover. Support the crucible cover on a clay triangle on an iron ring and heat the crucible cover gently until one of the two substances melts. (8) *Which has the lower melting point, sodium chloride or naphthalene?* Then heat the crucible cover more strongly for a few minutes. The molten solid will probably catch fire and burn with a smoky flame (for this reason, only a few crystals of each solid should be used). (9) *Does the other solid melt at the higher temperatures reached by the burner?*

E. Divide the remaining crystals of sodium chloride evenly between two test tubes. Divide the remaining crystals of naphthalene between two other test tubes. You should have nearly 0.1 g of solid in each test tube. To one test tube containing sodium chloride add 3 cm<sup>3</sup> of water and shake vigorously. (10) *Is sodium chloride soluble in water?* To one test tube containing naphthalene add 3 cm<sup>3</sup> of water and shake vigorously. (11) *Is naphthalene soluble in water?* To each of the two remaining test tubes add 3 cm<sup>3</sup> of benzene. (12) *Is sodium chloride soluble in benzene?* (13) *Is naphthalene soluble in benzene?*

### **Supplementary Questions:**

Prepare a chart, similar to the one below, where you can summarize your observations.

Physical Property	Sodium Chloride	Naphthalene
Odor (volatility)		
Hardness		
Melting point		
Solubility in water		
Solubility in benzene		
Bond type		

- (14) *How does bond type affect the volatility of a substance?*
- (15) *How does bond type affect the hardness of a solid?*
- (16) *How does bond type affect the melting point of a solid?*
- (17) *How does bond type affect the solubility of a substance in water?*
- (18) *How does bond type affect the solubility of a substance in benzene?*

# HYDROGEN CHLORIDE— A MOLECULE WITH A POLAR COVALENT BOND

## Purpose:

To prepare hydrogen chloride and to investigate the properties of a solution of hydrogen chloride in water.

## Introduction:

Hydrogen chloride may be prepared by reacting an acid such as sulfuric acid ( $H_2SO_4$ ) with a chloride such as sodium chloride (NaCl). CAUTION: Concentrated sulfuric acid is very corrosive to skin and clothing, and hydrogen chloride is very irritating to the mucous membrane. Do not prepare more than is absolutely necessary.

## Apparatus:

250 cm <sup>3</sup> Florence flask	ring stand
two-hole rubber stopper for flask	iron ring
bent glass tubes (2)	burner
rubber connector	wire gauze
thistle tube	buret clamp
wide-mouthed bottle	1000 cm <sup>3</sup> beaker
150 mm test tubes (7)	watch glass
rubber stopper for test tube	stirring rod
50 cm <sup>3</sup> graduated cylinder	

## Materials:

sodium chloride	mossy zinc
12 M sulfuric acid	copper turnings
red litmus paper	0.1 M silver nitrate
blue litmus paper	15 M ammonia water
magnesium ribbon	3 M nitric acid
iron filings	0.5 M sodium chloride

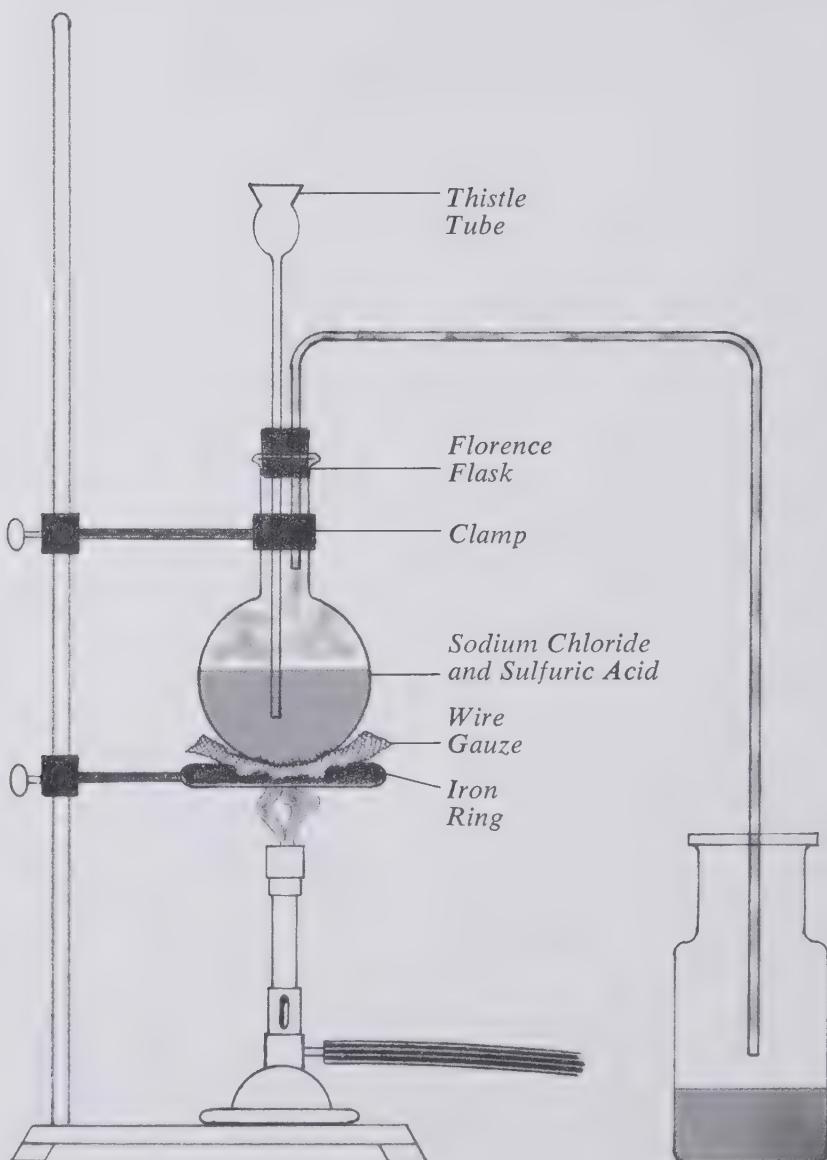
## Experiment:

- Assemble the apparatus as shown in Fig. 14-1. Place 10 g of sodium chloride in the flask and add 20 cm<sup>3</sup> of sulfuric acid through the thistle tube. Warm the flask gently and collect the gas which is produced in a dry test tube. Stopper

the test tube. (1) *What physical properties of hydrogen chloride have you observed?*

B. Place 30 cm<sup>3</sup> of water in a wide-mouthed bottle. Allow the hydrogen chloride to pass above the water for several minutes, but be careful to keep the end of the delivery tube above the level of the water. (2) *Why must the delivery tube not touch the water surface?* (3) *Is there any evidence that the hydrogen chloride is dissolving in the water?* Retain this liquid for use in parts D, E, and F.

C. Invert the test tube of hydrogen chloride gas in a 1000 cm<sup>3</sup> beaker which is half-full of water. Rapidly remove the



**Fig. 14-1** Apparatus for the Preparation of Hydrogen Chloride

stopper while the mouth of the tube is below the water surface. (4) *What can you say about the solubility of hydrogen chloride in water?* (5) *Do you suppose the nature of the bonding in hydrogen chloride has any effect on the solubility of hydrogen chloride in water, and if so, what is the effect?*

- D.** With a stirring rod, transfer one drop of the liquid from part B to a piece of blue litmus paper and a piece of red litmus paper placed on a watch glass. (6) *Does the liquid from part B have any effect on the red or on the blue litmus paper?*
- E.** Fill four test tubes one-half full with the liquid prepared in part B. In separate test tubes place magnesium ribbon, iron filings, mossy zinc, and copper turnings. (7) *Does the liquid have any effect on magnesium, iron, zinc, or copper?* (8) *In which test tube does the liquid have the greatest effect? The least effect?*
- F.** To 5 cm<sup>3</sup> of the liquid prepared in part B, add four drops of silver nitrate solution. (9) *What do you observe?* Add ammonia water dropwise until the reaction which occurs is complete. (10) *What do you observe?* Now cancel out the effect of the ammonia water by adding nitric acid until a drop of the solution causes the litmus paper to turn from blue to red. (11) *What do you observe?* Repeat this series of reactions in another test tube with a 5 cm<sup>3</sup> portion of sodium chloride solution instead of the liquid prepared in part B. (12) *Is the sequence of observations similar to the observations listed for questions 9 to 11?* (13) *If they are not similar, how do the observations differ?* (14) *For what purpose could the series of reactions in part F be used?*

# CARBON DIOXIDE— A DOUBLE-BONDED MOLECULE

## Purpose:

To prepare carbon dioxide and examine some of its properties.

## Introduction:

In this experiment carbon dioxide is prepared by reacting calcium carbonate with hydrochloric acid. The products are calcium chloride, water, and carbon dioxide. Like oxygen, carbon dioxide can be collected by displacement of water.

## Apparatus:

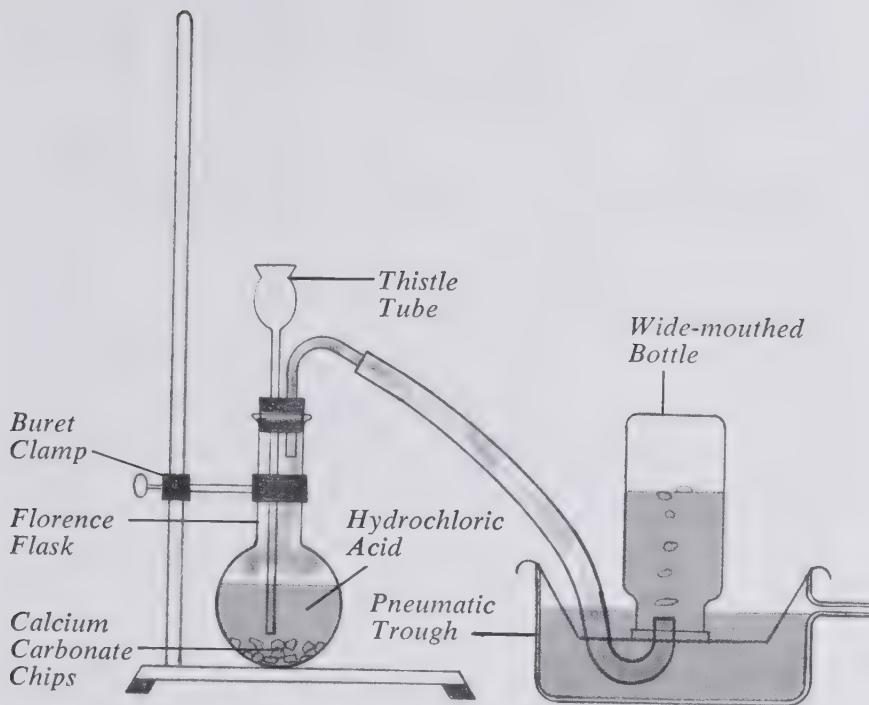
250 cm <sup>3</sup> Florence flask	wide-mouthed bottles (4)
two-hole rubber stopper	pneumatic trough
bent glass tube (60°)	buret clamp
thistle tube	ring stand
piece of rubber tubing	glass squares (4)
250 cm <sup>3</sup> beaker	deflagrating spoon
burner	150 mm test tube

## Materials:

calcium carbonate chips	sulfur
6 M hydrochloric acid	magnesium ribbon
wooden splint	candle
limewater	

## Experiment:

- A.** Place 50 g of calcium carbonate chips in an empty 250 cm<sup>3</sup> Florence or Erlenmeyer flask. A flask containing 50 g of chips is on display. Cover the calcium carbonate chips with water.
- B.** Set up the apparatus for the preparation and collection of carbon dioxide as in Fig. 15-1. Place four water-filled, wide-mouthed bottles upside down in a trough of water.
- C.** Carefully add hydrochloric acid a little at a time through the thistle tube. Maintain a constant evolution of gas for



**Fig. 15-1** Apparatus for the Preparation and Collection of Carbon Dioxide

about 15 s and then collect four bottles of carbon dioxide gas. Cover the mouth of each bottle with a glass square and remove the bottles from the trough. (1) *Is carbon dioxide very soluble in water?*

- D.** Light a wooden splint and thrust it into one bottle of carbon dioxide. (2) *Does the wooden splint continue to burn?*
- E.** Place a piece of sulfur about the size of a pea in a deflagrating spoon and light it in your Bunsen burner flame. Quickly lower the deflagrating spoon into a second bottle of carbon dioxide. (3) *Does the sulfur continue to burn?*
- F.** Wind the end of a 5 cm piece of magnesium ribbon around the bottom of a deflagrating spoon. Light the other end of the ribbon in your Bunsen burner flame. Do not look directly at the burning magnesium. Quickly lower the burning magnesium into a third bottle of carbon dioxide. (4) *Does the magnesium continue to burn?*
- G.** Place a short candle in a 250 cm<sup>3</sup> beaker and light it. Place the fourth bottle of carbon dioxide above the beaker. Tilt the bottle as if you were pouring water from the bottle into the beaker. Be careful not to pour any residual water from

the bottle onto the flame of the candle. (5) *Does the candle continue to burn?* (6) *What physical property of carbon dioxide is demonstrated?*

**H.** Bubble carbon dioxide from the generator into a test tube containing limewater. The result is a test for carbon dioxide. (7) *What is the result?*

### **Supplementary Questions:**

(8) *Was there any evidence that carbon dioxide had decomposed in any of parts D, E, or F? What was the evidence?*

(9) *Is carbon dioxide a stable molecule? What evidence do you have to support your answer?*

# THE DEGREE OF SATURATION OF A SOLUTION

## Purpose:

To prepare aqueous solutions of sodium acetate with different degrees of saturation and to study the properties of these solutions.

## Introduction:

Three possibilities can exist when a solute is dissolved in a given amount of solvent. The resulting solution can contain less solute than the solvent is capable of holding (be unsaturated); or it can contain the maximum amount of solute that the solvent is capable of holding (be saturated); or it can contain more than the maximum amount of solute that the solvent is capable of holding under the given conditions (be supersaturated).

In this experiment you will prepare solutions of each type, identify them, and study some of their properties.

## Apparatus:

10 cm <sup>3</sup> graduated cylinder	test tube holder
150 mm test tubes (2)	burner
cork stopper	250 cm <sup>3</sup> beaker

## Materials:

hydrated sodium acetate

## Experiment:

- A. Place 5 cm<sup>3</sup> of water in a 150 mm test tube. Add a few milligrams (a few tiny crystals) of hydrated sodium acetate and shake vigorously. (1) *What do you observe?* Add a few more crystals and shake again. (2) *What do you observe?* (3) *What kind of solution do you have?* (4) *How do you know?*
  
- B. Now add 3.5 g of hydrated sodium acetate to the solution. Stopper the test tube with a cork and shake vigorously for 1 min. (5) *What do you observe?* Allow the test tube to rest at an angle for a minute to promote faster settling of any suspended matter, then pour the clear upper liquid into

a dry test tube. To the clear liquid add a few milligrams of sodium acetate and shake vigorously for 1 min. (6) *What do you observe?* (7) *What kind of solution do you have?* (8) *How do you know?* (9) *Why did your second test tube have to be dry?*

- C. Pour the contents of the second tube back into the first tube and add an extra 3.0 g of hydrated sodium acetate. The added solid will form a plug just above the surface of the liquid. Holding the tube in a nearly horizontal position and taking care not to point the open end at anyone, heat the liquid gently so as to dissolve this plug partially. Then, holding the tube in a nearly upright position, heat to a gentle boil to dissolve all the crystals. Avoid the bumping which becomes more serious once all the crystals have dissolved.
- D. Gently place the test tube in a beaker of cold water and allow it to stand undisturbed for 5 min. If crystals appear at any point, remove the test tube, reheat it gently to redissolve the crystals and place the test tube in cold water for another 5 min. Gently remove the test tube and add one or two tiny crystals of sodium acetate. (10) *What do you observe?* (11) *What kind of solution did you have before the crystals were added?* (12) *How do you know?* (13) *What kind of solution did you have after the crystals were added?*

### **Supplementary Questions:**

- (14) *What happens when a small particle of solute is added to an unsaturated solution?*
- (15) *What happens when a small particle of solute is added to a saturated solution?*
- (16) *What happens when a small particle of solute is added to a supersaturated solution?*

# SOLUTIONS AND MOLECULAR POLARITY

## Purpose:

To study the effects of molecular polarity on the solubility of a substance in a liquid solvent.

## Introduction:

Most people know that sugar dissolves in some liquids but not in others. What factors determine whether one substance will dissolve in another? The solubility of one substance in another would depend on the nature of the two substances involved. For example, there are attractive forces between the molecules of sugar in a sugar crystal, just as there are attractive forces between the water molecules in pure water or, indeed, between the molecules of any pure substance. If sugar is going to dissolve in water, then the water must be able to overcome the attractive forces between the sugar molecules. If these forces are large, then the attractive forces between water molecules and sugar molecules must also be large. Thus the polarities of both the substance being dissolved and the solvent are important in determining whether or not a solution will result.

In this experiment you will attempt to dissolve nonpolar, polar, and ionic substances in solvents of different polarity. The solvents you will use are carbon tetrachloride (nonpolar), ethanol (polar), and water (highly polar). From the results you will formulate some principles on the effect of molecular polarity on the solubility of a substance in a solvent.

## Apparatus:

10 cm<sup>3</sup> graduated cylinder  
150 mm test tubes (9)

## Materials:

ethanol	ammonium chloride (NH <sub>4</sub> Cl)
carbon tetrachloride	glycerol
iodine (I <sub>2</sub> )	

## Experiment:

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them.

DATA TABLE	Solubility of I <sub>2</sub>	Solubility of NH <sub>4</sub> Cl	Solubility of Glycerol
Water			
Ethanol			
Carbon tetrachloride			

**B.** Place 5 cm<sup>3</sup> of water in a test tube. Using the level of water in the test tube as a guide, pour 5 cm<sup>3</sup> of ethanol and carbon tetrachloride separately into two other test tubes. (CAUTION: The vapors of carbon tetrachloride are poisonous. Avoid breathing these vapors. If possible, dispose of used carbon tetrachloride in a waste bottle in the fume hood.) To each of the three test tubes add two small iodine crystals. Stopper the test tubes and shake well. Observe the contents of each tube carefully to determine whether the iodine has dissolved. (1) *From your knowledge of the periodic table, electronegativities, and bond types, would you say that iodine molecules are nonpolar, polar, or ionic?* (2) *In which type of solvent does the iodine dissolve more readily?* (3) *How do you explain the differences you observed between the solubilities of iodine in water and in carbon tetrachloride?*

**C.** Using your test tube containing 5 cm<sup>3</sup> of water as a guide, pour separately into three more test tubes 5 cm<sup>3</sup> volumes of water, ethanol, and carbon tetrachloride. To each of the test tubes add two small crystals of ammonium chloride. Stopper the test tubes and shake vigorously. Observe the contents of each tube carefully to determine whether the ammonium chloride has dissolved. (4) *Is ammonium chloride a nonpolar, polar, or ionic substance?* (5) *In which type of solvent does the ammonium chloride dissolve more readily?* (6) *How do you explain, in terms of molecular polarities, the differences you observed between the solubilities of ammonium chloride in water and in carbon tetrachloride?*

**D.** Pour separately into three more test tubes, using your test tube containing 5 cm<sup>3</sup> of water as a guide, 5 cm<sup>3</sup> of water, ethanol, and carbon tetrachloride. To each of the test tubes add two drops of glycerol (C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>). Stopper the tubes and shake vigorously. Observe the contents of the tubes carefully to determine whether the glycerol has dissolved. (7) *Is glycerol a nonpolar, polar, or ionic substance?* (8) *In which*

*type of solvent does glycerol dissolve most readily? (9) How do you explain, in terms of molecular polarities, the differences you observed among the solubilities of glycerol in the three solvents?*

### **Supplementary Questions:**

- (10) *In what type of solvent is an ionic substance most soluble?*
- (11) *In what type of solvent is a polar substance most soluble?*
- (12) *In what type of solvent is a nonpolar substance most soluble?*
- (13) *What generalized statement, if any, can you make summarizing the effect of bond polarity on the solubility of a substance in a solvent?*
- (14) *When water and chloroform are mixed, the components separate into two layers, each of which smells strongly of chloroform. How would you quickly and easily confirm which layer is mainly water and which is mainly chloroform?*

# ACIDS AND BASES

## Purpose:

To discover some of the properties of acids and bases.

## Introduction:

Acids ionize in water, producing hydronium ions ( $\text{H}_3\text{O}^+$ ) and various negative ions. For example, nitric acid ionizes in water to produce hydronium ions and nitrate ions. Bases produce hydroxide ions ( $\text{OH}^-$ ) and various positive ions when they are dissolved in water. For example, potassium hydroxide produces potassium ions and hydroxide ions when it is dissolved in water.

The pH scale can be used to assess the acidity or basicity of a solution. Acidic solutions have pH values below seven. The further below seven the pH is, the more acidic the solution is. Basic solutions have pH values above seven. The further above seven the pH is, the more basic the solution is.

Acid-base indicators are substances which change color over different pH ranges. Methyl orange is red below pH 3.1 and orange-yellow above pH 4.4. Indigo carmine is blue in all solutions having a pH below 11.6, but it is yellow in solutions having a pH above 13. Phenolphthalein is colorless below pH 8.2 and turns pink above 8.2.

We will use this information regarding the pH scale and acid-base indicators to discover some properties of acids and bases.

## Apparatus:

watch glass	10 cm <sup>3</sup> graduated cylinder
stirring rods (2)	medicine dropper
150 mm test tubes (4)	

## Materials:

red litmus paper	methyl orange solution
blue litmus paper	indigo carmine solution
0.1 M hydrochloric acid	mossy zinc
0.1 M sodium hydroxide	6 M hydrochloric acid
0.1 M acetic acid	6 M acetic acid
0.1 M ammonia water	phenolphthalein solution

## Experiment :

A. Place a piece of red litmus paper and a piece of blue litmus paper on a watch glass. Using a stirring rod, transfer a drop of 0.1 M hydrochloric acid to one end of the piece of red litmus paper, and transfer a second drop to one end of the piece of blue litmus paper. (1) *What do you observe?* Now use another stirring rod to transfer one drop of 0.1 M sodium hydroxide to the other end (the dry end) of the piece of red litmus paper, and transfer a second drop to the other end of the piece of blue litmus paper. (2) *What do you observe?* (3) *For what purpose can litmus paper be used?*

B. Add 2 cm<sup>3</sup> of 0.1 M hydrochloric acid to a test tube containing 5 cm<sup>3</sup> of water. Add a drop of methyl orange indicator. Mix the solution. (4) *What do you observe?* Repeat, using 0.1 M acetic acid in place of the hydrochloric acid. (5) *What do you observe?* (6) *Do solutions of hydrochloric acid and acetic acid (both having the same concentration) appear to contain the same quantity of hydronium ions per unit volume?*

C. Place 5 cm<sup>3</sup> of 0.1 M sodium hydroxide in a test tube. Add two drops of indigo carmine indicator and mix the liquid in the test tube. (7) *What do you observe?* Repeat, using 0.1 M ammonia water. (8) *What do you observe?* (9) *Do solutions of sodium hydroxide and ammonia water (both having the same concentration) appear to contain the same quantity of hydroxide ions per unit volume?*

D. Add 1 cm<sup>3</sup> of 0.1 M sodium hydroxide to 9 cm<sup>3</sup> of water to form 0.01 M sodium hydroxide. Add two drops of indigo carmine solution to 5 cm<sup>3</sup> of the 0.01 M sodium hydroxide and mix the liquid in the test tube. (10) *Is the color of the indigo carmine the same as it was when you tested 0.1 M sodium hydroxide?* Add 1 cm<sup>3</sup> of 0.01 M sodium hydroxide to 9 cm<sup>3</sup> of water to form 0.001 M sodium hydroxide. Add two drops of indigo carmine solution to 5 cm<sup>3</sup> of the 0.001 M sodium hydroxide and mix the liquid in the test tube. (11) *Is the color of the indigo carmine the same as it was when you tested 0.01 M sodium hydroxide?* (12) *What is the effect of diluting the sodium hydroxide on the basicity of the solution?*

**E.** Place a small piece of mossy zinc in each of two test tubes. To the first test tube add  $5\text{ cm}^3$  of 6 M hydrochloric acid. (13) *What do you observe?* To the second test tube add  $5\text{ cm}^3$  of 6 M acetic acid. (14) *What do you observe?* (15) *Do the results of part E confirm your answer to question 6?*

**F.** Using a clean medicine dropper, place 20 drops of 0.1 M hydrochloric acid in a test tube. Rinse the medicine dropper with water and place 20 drops of 0.1 M acetic acid in a second test tube. Add one drop of phenolphthalein solution to each test tube. Rinse the dropper with water and add 0.1 M sodium hydroxide one drop at a time to the hydrochloric acid solution until you observe a change. (16) *What change do you observe?* (17) *What does this change indicate?* (18) *How many drops of sodium hydroxide were required to bring about the change?* Repeat this process, adding sodium hydroxide to the test tube containing the acetic acid until you observe a change. (19) *What change do you observe?* (20) *Is the change similar to the change recorded in question 16?* (21) *What does this change indicate?* (22) *How many drops of sodium hydroxide are required to bring about this change?* (23) *How do the answers to questions 18 and 22 compare?* (24) *What is the reason for this result?*

### **Supplementary Question:**

(25) *Commercial vinegar is a solution of acetic acid in water. How would you determine which brand of vinegar contains more acetic acid in a given volume of vinegar?*

# FORMING PRECIPITATES AND BALANCING EQUATIONS

## Purpose:

To prepare several precipitates and to balance the chemical equations for the reactions involved.

## Introduction:

Many compounds dissolve in water—they are soluble in water. However, other compounds are insoluble in water. When they are formed in water they immediately become solid precipitates. The formation of precipitates is frequently used by chemists in analytical work. For example, if a substance contains an unknown quantity of sulfate ions, the substance is first dissolved in water and then a reagent such as barium chloride is added to the solution. A precipitate of barium sulfate forms. Essentially all of the sulfate ions present in the substance are removed from the solution as the barium sulfate precipitate forms. The quantity of barium sulfate precipitate can be determined, and this information leads to a calculation of the amount of sulfate which must have been present in the sample.

One example of an insoluble compound is silver chloride. Silver nitrate and sodium chloride both dissolve in water, but when a solution of silver nitrate is mixed with a solution of sodium chloride, one of the products (silver chloride) is insoluble in water. The silver chloride forms a precipitate which falls to the bottom of the reaction container. The other product (sodium nitrate) remains dissolved.

Chemists do not generally like to write a paragraph to describe a chemical process if they can do it just as well in one line. A chemical equation is a convenient way of describing a chemical reaction. In the case of the preceding example, the chemical equation is:



This equation tells us that a solution of silver nitrate in water can be mixed with a solution of sodium chloride in water to form a solid precipitate of silver chloride and a solution of sodium nitrate in water.

## Apparatus:

150 mm test tubes (6)

## Materials:

lead nitrate  
potassium iodide  
iron(III) nitrate

potassium chromate  
calcium nitrate  
ammonium oxalate

## Experiment:

- A.** Take a small quantity of powdered lead nitrate (about the size of a pea) and dissolve it in water in a test tube. Use the smallest amount of water possible. In a second test tube dissolve a small quantity of potassium iodide in the least amount of water possible. (1) *What are the colors of the two solutions?* Mix the two solutions and observe the lead iodide precipitate. (2) *What is the color of the precipitate?*
- B.** In separate test tubes dissolve small quantities of iron(III) nitrate and potassium chromate in the smallest amounts of water possible. (3) *What are the colors of the two solutions?* Mix the two solutions and observe the iron(III) chromate precipitate. (4) *What is the color of the precipitate?*
- C.** In separate test tubes dissolve small quantities of calcium nitrate and ammonium oxalate in the smallest amounts of water possible. (5) *What are the colors of the two solutions?* Mix the two solutions and observe the calcium oxalate precipitate. (6) *What is the color of the precipitate?*
- D.** Write balanced chemical equations for each precipitation reaction. You must write correct chemical formulas for the compounds involved in the reactions, and you must make sure that you have the same number of atoms of each element on both sides of the yields sign. (7) *What does (aq) signify in a chemical equation?* (8) *What does (s) signify in a chemical equation?*

# TYPES OF CHEMICAL REACTION

## Purpose:

To study four different types of chemical reaction.

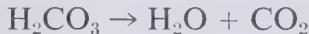
## Introduction:

It is not easy to classify all chemical reactions precisely. Nevertheless, most reactions can be classified into one of four major categories.

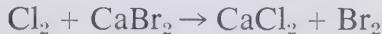
*Addition reactions*, or *direct combinations*, are reactions in which atoms and molecules join together directly to produce larger molecules. An example of an addition reaction is the combustion of sulfur to form sulfur dioxide:



*Decomposition reactions* are just the opposite of addition reactions. An example is the decomposition of carbonic acid:



*Displacement or substitution reactions* involve a change of partners. An example of a displacement reaction is the liberation of bromine from calcium bromide by the action of chlorine:



*Double decomposition or metathetic reactions* involve a joint exchange of partners, as in the precipitation of silver chloride when solutions of silver nitrate and sodium chloride are mixed:



In this experiment you will study eight different chemical reactions (one of them in a demonstration by the teacher). You will identify some of the products and then classify each reaction as one of the four types.

## Apparatus:

burner

medicine dropper

crucible tongs

10 cm<sup>3</sup> graduated cylinder

150 mm test tubes (5)

## Materials:

mercury(II) oxide	iron nail
wood splints	0.3 M copper sulfate
0.5 M sodium sulfate	copper wire
0.5 M barium chloride	6% hydrogen peroxide
magnesium ribbon	manganese dioxide
mossy zinc	iron(II) sulfide
2 M sulfuric acid	3 M hydrochloric acid

## Experiment:

A. *Teacher demonstration.* Heat a test tube containing some mercury(II) oxide. Insert a glowing splint into the tube.  
(1) *What do you observe?* Examine the contents of the tube.  
(2) *What do you observe?* (3) *Is a gas given off when the tube is heated?* (4) *If so, what is the gas?* (5) *What is the substance remaining in the test tube after the heating?* (6) *What word equation describes the reaction?* (7) *What type of chemical reaction is illustrated?*

B. Place 5 cm<sup>3</sup> of sodium sulfate solution in a test tube. Add a medicine dropper full of barium chloride solution. (8) *What do you observe?* (9) *If one of the products is sodium chloride (which is soluble in water), what is the other product?* (10) *What word equation describes the reaction?* (11) *What type of chemical reaction is illustrated?*

C. Hold a 3 cm strip of magnesium ribbon with a pair of crucible tongs. Insert the ribbon into a burner flame. Do not look directly at the ribbon while you are doing this. (12) *What do you observe?* (13) *With what is the magnesium combining?* (14) *What word equation describes the reaction?* (15) *What type of chemical reaction is illustrated?*

D. Place a small piece of mossy zinc in a test tube. Add 5 cm<sup>3</sup> of dilute sulfuric acid. (16) *What do you observe?* Insert a burning splint into the mouth of the tube. (17) *What do you observe?* (18) *What substance must be present?* (19) *What word equation describes the reaction?* (20) *What type of chemical reaction is illustrated?*

E. Place a clean iron nail in a test tube and cover it with a solution of copper sulfate. After several minutes remove the nail and examine it. (21) *Do you observe any change in the appearance of the nail?* (22) *If so, what is the change?*

(23) Do you observe any change in the appearance of the copper sulfate solution? (24) If so, what is the change? (25) What word equation describes the reaction? (26) What type of chemical reaction is illustrated?

**F.** Using crucible tongs, hold a 5 cm piece of copper wire in a burner flame. Remove the wire and examine it. (27) What do you observe? (28) With what did the copper combine? (29) What word equation describes this reaction? (30) What type of chemical reaction is illustrated?

**G.** Place 3 cm<sup>3</sup> of hydrogen peroxide in a test tube. Add a pinch of manganese dioxide. (31) What do you observe? Insert a glowing splint into the mouth of the test tube. (32) What do you observe? (33) What substance has been formed? (34) Is the manganese dioxide used up in this reaction? (35) If not, what is its function? (36) If water is another product of this reaction, what word equation describes the reaction? (37) What type of chemical reaction is illustrated?

**H.** Place a very small piece of iron(II) sulfide in a test tube. Add enough dilute hydrochloric acid to cover the iron(II) sulfide. (38) What do you observe? (39) Do you notice any odor? Do not inhale the gas. (40) If so, what does the odor resemble? Important: as soon as you have made your observations, pour the liquid contents of the tube into the sink and flush the sink with plenty of running water. (41) If iron(II) chloride is a water-soluble product, what word equation describes the reaction? (42) What type of chemical reaction is illustrated?

**I.** Write balanced chemical equations for all reactions in this experiment.

# REDOX REACTIONS OF METALS AND NONMETALS

## Purpose:

- (a) To arrange the metals, copper, zinc, and lead in order of their relative abilities to donate electrons and to arrange the metal ions in order of their ability to accept electrons.
- (b) To arrange the halogens, bromine, chlorine, and iodine in order of their relative abilities to accept electrons and to arrange the halide ions in order of their ability to donate electrons.

## Introduction:

The loss of electrons from an atom, with a consequent increase in oxidation number, is called *oxidation*. The substance which loses electrons is said to be oxidized, and the substance which removes them is called the oxidizing agent.

The gain of electrons by an atom, with a consequent decrease in oxidation number, is called *reduction*. The substance which gains electrons is said to be reduced, and the substance which supplies them is called the reducing agent.

Oxidation-reduction reactions involve a competition of substances for electrons. Strong oxidizing agents have a strong tendency to accept electrons. Strong reducing agents have little attraction for the electrons they already possess. Thus, tin(II) ion is a stronger oxidizing agent than is nickel(II) ion because the reaction:



goes predominantly to the right. That is, tin ions have a greater ability to remove electrons from nickel ions. Also, nickel metal is a stronger reducing agent than is tin metal, because nickel releases its electrons more readily.

In this experiment you will perform tests with three metals and their ions. By noting which tests give positive results you will be able to arrange the metals and their ions according to their relative abilities to donate and accept electrons. A similar series of tests with halogens will allow you to arrange the halogens and the halide ions according to their relative abilities to accept and donate electrons.

## Apparatus:

150 mm test tubes (6)  
10 cm<sup>3</sup> graduated cylinder

## Materials:

copper metal	chlorine water
zinc metal	bromine water
lead metal	iodine solution
0.1 M zinc(II) nitrate	0.1 M potassium bromide
0.1 M lead(II) nitrate	0.1 M potassium chloride
0.1 M copper(II) nitrate	0.1 M potassium iodide
carbon tetrachloride	

## Experiment:

A. Place a small piece of copper metal in 3 cm<sup>3</sup> of a solution of zinc(II) nitrate. (1) *What do you observe?* (2) *What can you say about the ability of copper atoms to donate electrons to zinc ions?*

Place a small piece of copper metal in 3 cm<sup>3</sup> of a solution of lead(II) nitrate. (3) *What do you observe?* (4) *What can you say about the ability of copper atoms to donate electrons to lead ions?*

B. Similarly, test the behavior of small pieces of zinc metal in separate solutions of copper(II) nitrate and lead(II) nitrate. (5) *What do you observe in each case?* (6) *What can you say about the ability of zinc atoms to donate electrons to copper ions? To lead ions?*

C. Next, add small pieces of lead metal to separate solutions of copper(II) nitrate and zinc(II) nitrate. (7) *What do you observe in each case?* (8) *What can you say about the ability of lead metal to donate electrons to copper ions? To zinc ions?*

D. In each of three test tubes place 1 cm<sup>3</sup> of carbon tetrachloride and 3 cm<sup>3</sup> of water. (9) *What do you observe?* To one test tube add 5 drops of chlorine water and shake vigorously. (10) *What color is a solution of chlorine in water? In carbon tetrachloride?* To a second test tube add 5 drops of bromine water and shake vigorously. (11) *What color is a solution of bromine in water? In carbon tetrachloride?* (12) *In which solvent is the bromine more soluble?* To the third test tube

add 5 drops of iodine solution and shake vigorously. (13) *What is the color of a solution of iodine in water? In carbon tetrachloride? (14) In which solvent is the iodine more soluble?*

**E.** Test the behavior of a  $3\text{ cm}^3$  sample of potassium bromide solution with  $1\text{ cm}^3$  of carbon tetrachloride when 5 drops of chlorine water are added and shaken vigorously. Repeat, using 5 drops of iodine solution instead of the chlorine water. (15) *In each case, is there a color in either layer? (16) If so, in which layer is the color? (17) What do your results tell you about the ability of bromide ions to donate electrons to chlorine molecules? To iodine molecules?*

**F.** Repeat, testing  $3\text{ cm}^3$  portions of potassium chloride solution separately with bromine water and with iodine solution. Remember to add the  $1\text{ cm}^3$  of carbon tetrachloride. (18) *In each case, is there a color in either of the layers? (19) If so, in which layer is the color? (20) What do your results tell you about the ability of chloride ions to donate electrons to bromine molecules? To iodine molecules?*

**G.** Finally, test  $3\text{ cm}^3$  portions of potassium iodide solution separately with chlorine water and with bromine water. Remember to add the  $1\text{ cm}^3$  of carbon tetrachloride. (21) *In each case, is there a color in either of the layers? (22) If so, in which layer is the color? (23) What do your results tell you about the ability of iodine ions to donate electrons to chlorine molecules? To bromine molecules?*

## Supplementary Questions:

(24) *Write equations for each reaction that took place.*

(25) *Which metal is the strongest reducing agent? The weakest reducing agent?*

(26) *Which metal ion is the strongest oxidizing agent? The weakest oxidizing agent?*

(27) *Which halogen is the strongest oxidizing agent? The weakest oxidizing agent?*

(28) *Which halide ion is the strongest reducing agent? The weakest reducing agent?*

(29) *Prepare a table with two columns. In the first column arrange the three metals, with the strongest reducing agent*

at the top and the weakest reducing agent at the bottom. In the other column place the weakest oxidizing agent at the top and the strongest oxidizing agent at the bottom. What do you notice?

(30) Similarly, prepare a table of two columns for the halogens and the halide ions, placing the strongest reducing agent at the upper left, the weakest reducing agent at the lower left, the strongest oxidizing agent at the lower right, and the weakest oxidizing agent at the upper right. What do you notice? Is there any apparent relation between the oxidizing ability of a halogen and its position in the periodic table?

# IDENTIFICATION OF SOLUTIONS

## Purpose:

To identify five solutions.

## Introduction:

In this experiment, you are asked to identify the following solutions:

0.1 M $\text{H}_2\text{SO}_4$	0.1 M $\text{NaOH}$
0.1 M $\text{Ba}(\text{OH})_2$	0.1 M $\text{Na}_2\text{SO}_4$
0.1 M $\text{Pb}(\text{NO}_3)_2$	

The solutions are dispensed in coded test tubes. Your only tool for identification of the solution present in each test tube is a solution of methyl orange, an indicator which is pinkish-red in acid solution and yellowish-orange in neutral and basic solution. Of the five unknown solutions, only the sulfuric acid is acidic enough to turn the methyl orange pinkish-red. Sodium hydroxide and barium hydroxide are basic, and sodium sulfate and lead nitrate are neutral salts. If you were to add methyl orange to 5 cm<sup>3</sup> of each separate solution, you should be able to identify the sulfuric acid.

You can now use the sulfuric acid to test the other four solutions. Sulfuric acid will cause a precipitate of lead sulfate to form when it is added to a solution of lead nitrate. The solution will still be acidic because nitric acid is formed. Sulfuric acid will cause the formation of a precipitate of barium sulfate when it is added to barium hydroxide. The solution will no longer be acidic because the base (barium hydroxide) is neutralized by the sulfuric acid to form the precipitate and water.

Sulfuric acid does not react with sodium sulfate. There will be no precipitate and the solution will still be acidic because of the sulfuric acid. Sulfuric acid is neutralized by the sodium hydroxide but no precipitate is formed. The solution is not acidic because both the sodium sulfate and the water formed are neutral.

## Apparatus:

150 mm test tubes (5)  
10 cm<sup>3</sup> graduated cylinder

## Materials:

1 set of solutions in coded test tubes  
(e.g., A1 to A5, B1 to B5, etc.)  
methyl orange solution

## Experiment:

- A. Pour 5 cm<sup>3</sup> of each unknown solution into separate test tubes. Add 2 drops of methyl orange solution to each test tube. (1) *Which test tube contains the sulfuric acid?*
- B. Add 1 cm<sup>3</sup> of the sulfuric acid to each of the four remaining test tubes which contain 5 cm<sup>3</sup> of solution and 2 drops of methyl orange. Observe the color of the solution and note the presence or absence of a precipitate in each test tube. (2) *Which test tube contains the lead nitrate? (3) Which test tube contains the sodium sulfate? (5) Which test tube contains the sodium hydroxide?*
- C. Write balanced chemical equations for each reaction that occurs in this experiment.

# SOLUTIONS AND SOLUBILITY

## Purpose:

To identify six unknown solutions.

## Introduction:

In this experiment you are asked to identify six solutions. The solutions are:

0.1 M CuSO <sub>4</sub>	0.1 M Na <sub>2</sub> CO <sub>3</sub>
0.1 M Cu(NO <sub>3</sub> ) <sub>2</sub>	0.1 M (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>
0.1 M BaCl <sub>2</sub>	0.1 M ZnSO <sub>4</sub>

The solutions are dispensed in coded test tubes. Your major tool for identifying the solutions will be the following information:

All nitrates are soluble.

Nearly all compounds of lithium, sodium, potassium, and ammonium are soluble.

Nearly all chlorides are soluble except for AgCl, Hg<sub>2</sub>Cl<sub>2</sub>, and PbCl<sub>2</sub>.

Nearly all sulfates are soluble except for CaSO<sub>4</sub>, BaSO<sub>4</sub>, SrSO<sub>4</sub>, and PbSO<sub>4</sub>.

Nearly all carbonates are insoluble; however, Li<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, and (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> are soluble.

In addition, copper ions are blue, while all other ions in the unknown solutions are colorless.

There are many ways to identify the six solutions. One suggestion is given in the experimental procedure.

## Apparatus:

150 mm test tubes (14)  
10 cm<sup>3</sup> graduated cylinder

## Materials:

1 set of solutions in coded test tubes  
(e.g., A1 to A6, B1 to B6, etc.)

## Experiment:

- A. Take 10 cm<sup>3</sup> of each solution from the coded test tubes and give each solution a fixed position in your test tube rack. Use only a small portion of each unknown solution in any one test.
- B. Identify the copper solutions by their color. (1) *Which two coded test tubes contain copper solutions?*
- C. Add 1 cm<sup>3</sup> of each copper solution to 1 cm<sup>3</sup> of each of the other four unknown solutions, all in separate test tubes. (Note: A total of eight test tubes will be used.) Observe whether or not a precipitate forms in each case. (2) *Which mixtures of solutions produce precipitates?*
- D. To help you identify CuSO<sub>4</sub>, Cu(NO<sub>3</sub>)<sub>2</sub>, BaCl<sub>2</sub>, and Na<sub>2</sub>CO<sub>3</sub> solutions, note the following:
  - i) When CuSO<sub>4</sub> solution is added to each of the four solutions, precipitates will form with BaCl<sub>2</sub> and Na<sub>2</sub>CO<sub>3</sub>. The precipitates are barium sulfate and copper carbonate, respectively.
  - ii) When Cu(NO<sub>3</sub>)<sub>2</sub> is added to each of the four solutions, a precipitate will form only with Na<sub>2</sub>CO<sub>3</sub>. The precipitate is copper carbonate.(3) *Which test tube contains CuSO<sub>4</sub>?* (4) *Which test tube contains Cu(NO<sub>3</sub>)<sub>2</sub>?* (5) *Which test tube contains Na<sub>2</sub>CO<sub>3</sub>?* (6) *Which test tube contains BaCl<sub>2</sub>?*
- E. Devise a method to identify the ammonium sulfate (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and the zinc sulfate ZnSO<sub>4</sub>. (7) *What method did you use to distinguish between the (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and the ZnSO<sub>4</sub>?* (8) *Which test tube contains (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>?* (9) *Which test tube contains ZnSO<sub>4</sub>?*

## Supplementary Question:

- (10) *What is the balanced equation for each mixture of solution that gives a precipitate and what is the name of the precipitate in each case?*

# THE FORMULA OF A HYDRATE

## Purpose:

To determine the ratio of moles of water to moles of barium chloride in the hydrate  $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$ .

## Introduction:

When the water is evaporated from an aqueous solution of a salt, water often becomes incorporated into the crystals of salt that form. These crystals may appear to be dry, but they will yield a quantity of water when heated. Salts which contain water as part of their crystal structure are called *hydrates* and the water is called *water of hydration*.

In this experiment you will determine the number of moles of water combined with each mole of barium chloride in the hydrate  $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$ . When hydrated barium chloride is heated, water is given off:



We can determine the mass of water lost when a known mass of the hydrate is heated. We can use this information to calculate the number of moles of water lost and the number of moles of  $\text{BaCl}_2$  left behind. The mole ratio of water to  $\text{BaCl}_2$  can then be calculated.

## Apparatus:

crucible and cover  
clay triangle  
iron ring  
ring stand

burner  
wire gauze  
crucible tongs

## Materials:

hydrated barium chloride

## Experiment:

- A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

DATA TABLE	
Mass of crucible and cover	_____ g
Mass of crucible, cover, and $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$	_____ g
Mass of $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$	_____ g
Mass of crucible, cover, and $\text{BaCl}_2$ (first heating)	_____ g
Mass of crucible, cover, and $\text{BaCl}_2$ (second heating)	_____ g
Mass of $\text{BaCl}_2$	_____ g
Mass of $\text{H}_2\text{O}$ lost	_____ g
Moles of $\text{BaCl}_2$	_____ mol
Moles of water	_____ mol
Moles of water per mole of $\text{BaCl}_2$	_____ mol

**B.** Place a clean, dry crucible and cover on a triangle placed on a ring stand. The crucible cover should be slightly ajar to permit the escape of any water in the crucible. Heat the crucible with a burner for two or three minutes, then place the crucible and cover on a wire gauze to cool for about five minutes. Determine the mass of the crucible and cover.  
(1) *What is the purpose of this initial heating and cooling?*

**C.** Place enough  $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$  crystals in the crucible to fill it one-fourth full (about 4 g or 5 g of crystals). Determine the mass of the crucible, cover, and crystals.

**D.** Place the crucible, with its cover slightly ajar, on the triangle. Heat very gently for about five minutes or until hissing or sizzling has stopped. Avoid spattering the contents of the crucible. Increase the flame until the crucible bottom is a dull red, and heat for five minutes. Place the crucible and cover on a wire gauze to cool for about five minutes, then determine the mass of the crucible, cover, and contents.

**E.** Reheat the crucible and contents to dull redness for about three minutes, cool, and redetermine the mass. Your measurements from parts D and E should agree within 0.03 g. If they do not agree, repeat part E. (2) *What is the purpose of this second heating and cooling?* (3) *What is the formula for hydrated barium chloride?*

# EMPIRICAL FORMULA OF A COMPOUND

## Purpose:

To determine the empirical formula of a compound of magnesium and oxygen.

## Introduction:

In this experiment you will produce a compound of magnesium and oxygen by heating magnesium in the presence of air. Some of the magnesium reacts with nitrogen in the air to form a compound of magnesium and nitrogen. This is converted to the compound of magnesium and oxygen by adding water and reheating the solid.

## Apparatus:

crucible and cover	burner
clay triangle	crucible tongs
iron ring	medicine dropper
ring stand	wire gauze

## Materials:

magnesium ribbon

## Experiment:

- A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.
- B. Determine the mass of a clean, dry crucible and cover to the nearest 0.01 g.
- C. Cut a 40 cm piece of magnesium ribbon into two pieces of approximately equal length. Roll each piece into a loose coil and place both coils in the bottom of the crucible. Determine the mass of the crucible, cover, and magnesium coils.
- D. Place the covered crucible and contents on a triangle supported on an iron ring clamped to a ring stand. The cover

## DATA TABLE

Mass of crucible and cover	_____ g
Mass of crucible, cover, and magnesium	_____ g
Mass of magnesium	_____ g
Mass of crucible, cover, and oxide of magnesium	_____ g
Mass of oxygen contained in the oxide of magnesium	_____ g
Moles of magnesium atoms	_____ mol
Moles of oxygen atoms	_____ mol
Ratio of moles of magnesium to moles of oxygen	_____
Empirical formula of the oxide of magnesium	_____

should be adjusted so that it is slightly ajar to allow limited access of air. Heat very gently so that the reaction does not smoke. If the reaction becomes too vigorous, close the cover temporarily to slow down the reaction. This process should take about five minutes. (1) *Why must you take precautions to prevent the loss of a large amount of smoke?*

- E.** Heat for another five minutes, then remove the cover completely and heat as strongly as possible for another five minutes. Allow to cool for about three minutes.
- F.** Carefully add about 10 drops of water making sure that all of the solid is wetted. Carefully smell the moist solid. (2) *Can you recognize any odor? (3) What reaction produces this odor?*
- G.** Warm gently to allow the water to evaporate without spattering. Cover the crucible and heat strongly for ten minutes, then allow to cool to near room temperature. Determine the mass of the crucible, cover, and oxide of magnesium.

### Supplementary Question:

- (4) *If you had accidentally lost some material due to spattering in part G, would your apparent ratio of moles of Mg atoms to moles of O atoms have been too high or too low? Explain.*

# MASS CHANGES DURING A CHEMICAL REACTION— A TWO-PERIOD EXPERIMENT

## Purpose:

To investigate the mass changes which occur during the reaction between zinc metal and lead acetate.

## Introduction:

In this experiment a strip of zinc metal will be placed in an aqueous solution of lead acetate. The reaction will be allowed to proceed for at least 24 h, and mass changes which occur during the reaction will be determined. This information will be used to calculate the mole ratios involved in the chemical reaction between zinc metal and lead acetate.

## Apparatus:

250 cm<sup>3</sup> beakers (2)  
vial and cover  
100 cm<sup>3</sup> graduated cylinder  
stirring rod with rubber policeman  
watch glass  
wash bottle

## Materials:

lead acetate	glacial acetic acid
zinc strip	acetone

## Experiment:

- A.** Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.
  
- B.** Obtain a clean, dry 250 cm<sup>3</sup> beaker and determine its mass. Obtain a vial containing about two grams of lead acetate and determine its mass. (1) *What is the formula of lead acetate?* (2) *What is the molar mass of lead acetate?* Pour the solid lead acetate into the beaker. Determine the mass of the empty vial and cover.

## DATA TABLE

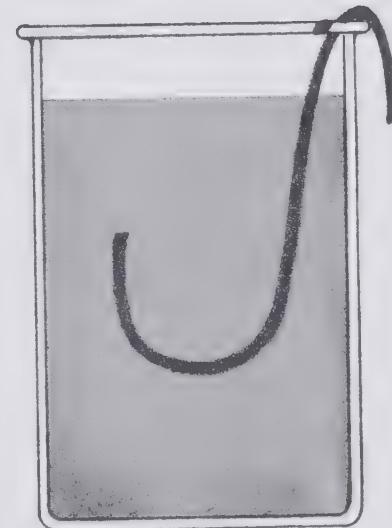
Mass of vial, cover, and lead acetate	_____ g
Mass of empty vial and cover	_____ g
Mass of lead acetate used	_____ g
Mass of zinc strip before reaction	_____ g
Mass of zinc strip after reaction	_____ g
Mass of zinc involved in reaction	_____ g
Mass of clean, dry $250\text{ cm}^3$ beaker	_____ g
Mass of beaker and lead crystals	_____ g
Mass of lead involved in reaction	_____ g
Moles of lead acetate used	_____ mol
Moles of zinc involved in reaction	_____ mol
Moles of lead involved in reaction	_____ mol

C. Measure  $200\text{ cm}^3$  of distilled water into the beaker and stir. (3) *What do you observe?* Add ten drops of glacial acetic acid to the liquid and stir again. (4) *What is the purpose of the acetic acid?* Stir the liquid until all of the lead acetate has dissolved. Using a wash bottle, wash any lead acetate solution, which adheres to the stirring rod, into the solution in the beaker.

D. Obtain a zinc strip and determine its mass. Place the zinc strip in the solution of lead acetate so that about 90% of the strip is in the solution and 10% of the strip extends out of the solution (Fig. 26-1). Cover the beaker with a watch glass. After several minutes observe the contents of the beaker. (5) *What do you observe?* Allow the reaction to proceed for at least 24 h.

E. Remove the watch glass, lift the zinc strip out of the solution, and hold the strip above the beaker. Using the stirring rod with a rubber policeman, scrape the lead metal off the zinc strip and allow the lead to fall into the beaker. Using a wash bottle rinse the zinc strip with water and allow the liquid to fall into the beaker.

F. Place the zinc strip in a second beaker and wash it with acetone. Allow the zinc strip to dry and determine its mass.



**Fig. 26-1** Zinc Strip in Lead Acetate Solution

**G.** Decant the liquid in the first beaker leaving the solid lead crystals in the beaker. Add 50 cm<sup>3</sup> of distilled water to the solid crystals in the beaker and decant this liquid. Add 25 cm<sup>3</sup> of acetone to the crystals and decant the acetone, leaving the lead in the beaker.

**H.** Dry the beaker and lead crystals using either a drying oven or a heat lamp, and determine the total mass of the beaker and lead crystals.

### **Supplementary Questions:**

- (6) *What is the ratio of moles of zinc to moles of lead involved in this reaction?*
- (7) *What is the ratio of moles of zinc to moles of lead acetate involved in this reaction?*
- (8) *What is the fourth substance involved in this reaction? What is its formula?*
- (9) *What is the balanced chemical equation which represents this reaction?*

# THE EFFECT OF PRESSURE ON THE VOLUME OF A GAS

27

## Purpose:

To discover the relationship between the volume of a given quantity of a gas and the pressure exerted on the gas.

## Introduction:

In this experiment a fixed quantity of a gas is trapped in a capillary tube by a column of mercury. Since the diameter of the tube is constant, the length of the trapped column of gas is directly proportional to the volume of the gas. We will define the volume of a column of gas having a length of 1.0 mm as 1.0 vol. Thus, if the length of the column is 2.0 mm, the volume will be 2.0 vol; if the length is 3.0 mm, the volume will be 3.0 vol; and so on. We will do this in order to avoid the apparent anomaly of measuring volume quantities in units of length.

The pressure exerted by the gas is equal to the sum of the pressures exerted on it by the atmosphere and by the mercury column. For example, when the tube is in a vertical upright position, the pressure exerted on the gas is the sum of the atmospheric pressure and that caused by the vertical height of the mercury. When the tube is in a horizontal position, the vertical height of the mercury is zero and the mercury column exerts no pressure on the trapped gas. Therefore, the gas pressure is the same as the atmospheric pressure. When the tube is in a vertical upside-down position, the mercury column tries to expand the gas, and the total pressure is equal to the atmospheric pressure minus that exerted by the vertical height of the mercury column.

When the tube is inclined at a  $45^\circ$  angle to the horizontal, the situation is somewhat more complicated because the vertical height of the mercury column ( $h$ ) is no longer the same as the length measured along the tube ( $\ell$ ). In such a situation it can be shown that  $h = 0.707\ell$  (i.e.,  $h = \ell \sin 45^\circ$ ). Thus, the pressure exerted on the gas equals the atmospheric pressure plus the pressure exerted by 0.707 times the length of the mercury column (Fig. 27-1).

## Apparatus:

capillary tube with a mercury column  
15 cm ruler  
masking tape

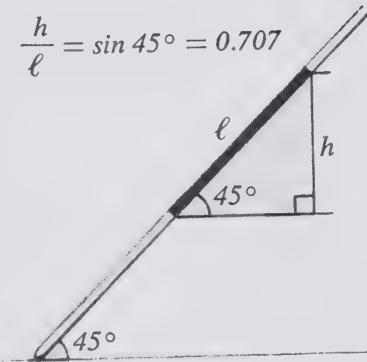


Fig. 27-1

## Experiment:

CAUTION: Mercury vapors are given off from the metal and are exceedingly dangerous. Although very little mercury is used in this experiment, be especially careful not to spill any of it. Inform your teacher immediately of any accident.

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

DATA TABLE			
Atmospheric pressure		_____ kPa	_____ mm
Position of tube	Volume of Gas (V)	Total Pressure (P)	PV Product
Horizontal	_____ vol	_____ kPa	_____
Vertical upright	_____ vol	_____ kPa	_____
Vertical upside-down	_____ vol	_____ kPa	_____
45° to horizontal	_____ vol	_____ kPa	_____
Other (e.g., 30° or 60°)	_____ vol	_____ kPa	_____

B. With the tube in a horizontal position on the desk, measure the length of the mercury column using a 15 cm ruler. The pressure (in kPa) exerted by a column of mercury is numerically equal to 0.133 times the vertical height of the column (in mm).

C. Using two pieces of masking tape, fasten the ruler to the capillary tube so that the 0 cm mark is at the bottom of the inner column of trapped air (not at the outside bottom of the glass tube itself) (Fig. 27-2).

D. With the tube again in the horizontal position on the desk, measure the length (i.e., volume) of the column of trapped air. Determine the pressure exerted on the air.

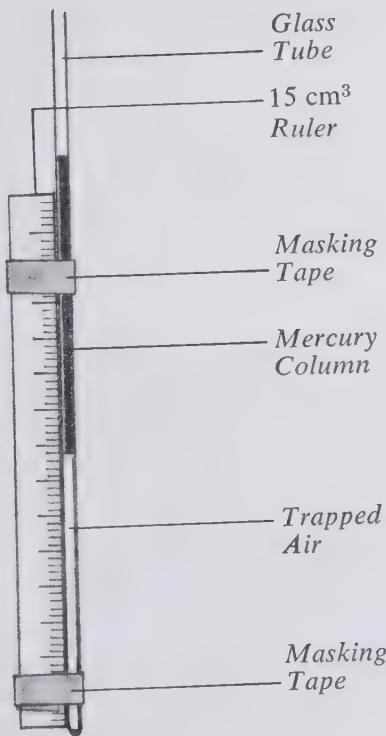
E. Place the tube in a vertical upright position, and again measure the length of the column of trapped air. Determine the pressure exerted on the air.

F. Carefully invert the tube and hold it in a vertical upside-down position. Measure the length of the column of trapped air. Determine the pressure exerted on the air.

G. Return the tube to a horizontal position and then incline it at an angle of  $45^\circ$  to the horizontal. Again measure the length of the column of trapped air. Determine the pressure exerted on the air. (1) *What is the effect of an increase of pressure on the volume of the trapped air?* (2) *What can you say about the constancy of the PV products?*

## Supplementary Questions:

- (3) *What type of graph is obtained if volume (vertical axis) is plotted against pressure (horizontal axis)?*
- (4) *What type of graph is obtained if volume (vertical axis) is plotted against  $1/\text{pressure}$  (i.e., the reciprocal of pressure)?*
- (5) *What is the relationship between the pressure and the volume of a given quantity of a gas at a constant temperature?*
- (6) *If the pressure on your quantity of air had been 130 kPa, what would have been the volume?*



**Fig. 27-2** Boyle's Law Apparatus

# THE EFFECT OF TEMPERATURE ON THE VOLUME OF A GAS

## Purpose:

To discover the relationship between the volume and the temperature of a given quantity of a gas.

## Introduction:

In this experiment a fixed quantity of a gas is trapped in a capillary tube by a short column of mercury. Since the diameter of the tube is constant, the length of the trapped column of gas is directly proportional to the volume of the gas. We will define the volume of a column of gas having a length of 1.0 mm as 1.0 vol. Thus, if the length of the column is 2.0 mm, the volume will be 2.0 vol; if the length is 3.0 mm, the volume will be 3.0 vol; and so on. We will do this to avoid the apparent contradiction of measuring volume quantities in units of length.

The temperature of the trapped gas can be varied by immersing the capillary tube in a beaker of water, the temperature of which can be adjusted to any desired point. The Kelvin temperature is obtained by adding 273° to the Celsius temperature.

## Apparatus:

15 cm ruler	thermometer
masking tape	600 cm <sup>3</sup> beaker
capillary tube with a mercury plug	

## Experiment:

**CAUTION:** Mercury vapors are exceedingly poisonous. Although very little mercury is used in this experiment, be especially careful not to spill any of it. Inform your teacher immediately of any accident.

- A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.
- B. Obtain a glass capillary tube containing a mercury plug.

## DATA TABLE

Tempera-ture ( $t$ )	Tempera-ture ( $T$ )	Volume	$V/t$ ratio	$V/T$ ratio
_____ °C	_____ K	_____ vol	_____	_____
_____ °C	_____ K	_____ vol	_____	_____
_____ °C	_____ K	_____ vol	_____	_____
_____ °C	_____ K	_____ vol	_____	_____
_____ °C	_____ K	_____ vol	_____	_____
_____ °C	_____ K	_____ vol	_____	_____

- C. Using two pieces of masking tape, fasten a 15 cm ruler to the glass tube so that the 0 cm mark is at the bottom of the inner column of trapped air (not at the outside bottom of the glass tube itself).
- D. Using two more pieces of masking tape, fasten the ruler and tube assembly to a thermometer so that the bottom of the tube is near the bottom of the thermometer (Fig. 28-1).
- E. Allow the tube to stand for a few minutes to come to room temperature. Record the room temperature and measure the height of the column of trapped air.
- F. Immerse the apparatus in a beaker containing enough cold water so that the column of air is completely covered. Record the temperature and measure the height of the air column.
- G. Adjust the temperature of the water in the beaker by adding hot water in four steps so as to obtain readings at four other temperatures ranging from colder to warmer. (Water from a hot water tap is probably hot enough to obtain a good range of temperatures.) Stir the water in the beaker to insure a uniform temperature. Measure the height of the column of trapped air for each temperature. (1) *What is the effect of an increase of temperature on the volume of the trapped air?* (2) *What can you say about the constancy of the  $V/t$  ratios?* (3) *What can you say about the constancy of the  $V/T$  ratios?*

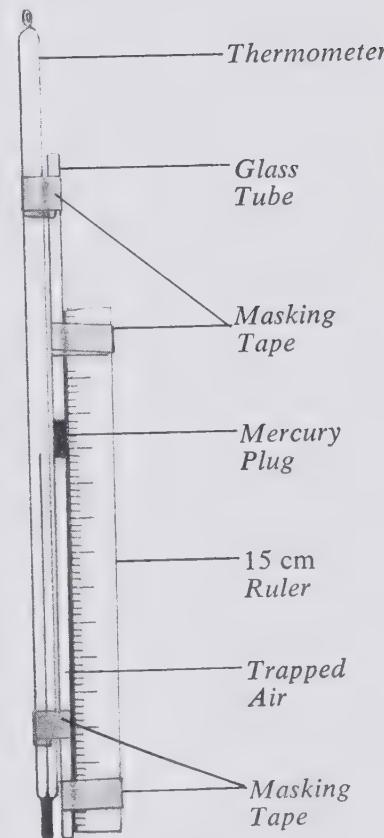


Fig. 28-1 Charles' Law Apparatus

## Supplementary Questions:

- (4) *Plot a graph of volume (vertical axis) against temperature (horizontal axis). Use the temperature scale (Celsius or Kelvin) which gives the most constant volume-to-temperature ratios. What type of graph is obtained?*
- (5) *Using the same temperature scale you used in question 4, plot a graph of volume against the reciprocal of temperature. What type of graph is obtained?*
- (6) *What is the relationship between the temperature and the volume of a given quantity of a gas at a constant pressure?*
- (7) *Why is the vol not a universal unit of measurement?*

# REACTION OF HYDROCHLORIC ACID WITH MAGNESIUM

29

## Purpose:

To determine the volume of hydrogen gas produced when a given quantity of magnesium reacts with excess hydrochloric acid.

## Introduction:

In this experiment a given mass of magnesium will be allowed to react with excess hydrochloric acid in a gas measuring tube. The volume of hydrogen produced will be measured and the room temperature and pressure will be recorded.

Since the hydrogen gas will be collected over water, the hydrogen will be mixed with water vapor. The pressure of the dry hydrogen gas can be calculated by subtracting the water vapor pressure from the room pressure. Water vapor pressures at various temperatures are found in Table 29-1.

TABLE 29-1 WATER VAPOR PRESSURE

°C	kPa	°C	kPa
15	1.70	23	2.81
16	1.82	24	2.98
17	1.94	25	3.17
18	2.06	26	3.36
19	2.20	27	3.57
20	2.34	28	3.78
21	2.49	29	4.00
22	2.64	30	4.24

Knowing the volume, pressure, and temperature of dry hydrogen gas, the volume can be corrected to STP. The mass of magnesium used can be converted to the number of moles of magnesium used. The volume of dry hydrogen at STP produced by one mole of magnesium can then be calculated.

## Apparatus:

metric ruler	10 cm <sup>3</sup> graduated cylinder
thread	one-hole rubber stopper, size 00
gas measuring tube	battery jar
ring stand	thermometer
buret clamp	barometer
400 cm <sup>3</sup> beaker	

## Materials:

magnesium ribbon
6 M hydrochloric acid

## Experiment:

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

DATA TABLE	
Mass of one metre of Mg	_____ g
Length of Mg used	_____ cm
Mass of Mg used	_____ g
Moles of Mg used	_____ mol
Room temperature	_____ °C
Room pressure (obtained from teacher)	_____ kPa
Temperature of the water	_____ °C
Volume of hydrogen gas and water vapor collected	_____ l

B. Obtain a piece of magnesium ribbon about 3-4 cm long. Measure this ribbon as precisely as possible with a metric ruler. The magnesium ribbon is assumed to be uniform in thickness and width. You will be told the mass of one metre of the ribbon. From this you can calculate the mass of your piece of ribbon.

C. Roll the piece of magnesium ribbon into a coil and tie it with a piece of thread about 20 cm long. The coil must be small enough to fit into the gas measuring tube.

D. Set up a ring stand and buret clamp, and fill a 400 cm<sup>3</sup> beaker about two-thirds full of water. Figure 29-1 shows what the apparatus will look like when the gas measuring tube is in position.

E. Carefully pour 10 cm<sup>3</sup> of 6 M hydrochloric acid into the gas measuring tube. Slowly fill the tube with tap water. Rinse down any acid that may be on the walls of the tube but try to avoid stirring up the acid layer in the bottom of the tube. The tube should be completely filled with water so that when a one-hole stopper (size 00) is placed in the tube a little water will be forced out. Any bubbles sticking to the walls of the tube may be dislodged by tapping the tube gently.

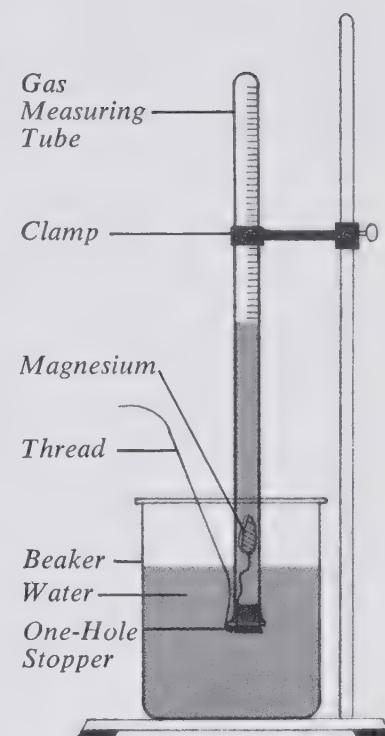
F. Holding the thread, lower the magnesium coil into the water to a depth of about 5 cm. Place the one-hole stopper into the mouth of the tube so that the thread is caught between the glass and the stopper.

G. Cover the hole in the stopper with your finger and invert the tube. Place the inverted tube in the beaker of water. Clamp the tube in position (Fig. 29-1). The hydrochloric acid will eventually reach the magnesium ribbon and reaction will occur.

H. When the magnesium ribbon has disappeared and the reaction has finished, wait for 5 min to allow the gas in the tube to come to room temperature. (1) *Why is this necessary?* Dislodge any gas bubbles that are sticking to the walls of the tube by tapping the tube.

I. Cover the hole in the stopper with your finger and transfer the tube to a battery jar or a large graduated cylinder filled with water. Remove your finger and adjust the tube so that the level of the water inside the tube is the same as the level of the water outside the tube. (2) *At this point what is the total pressure exerted by the gases in the tube?* (3) *What two gases are present in the tube?* Read the gas volume. Drain and clean the gas measuring tube.

J. Measure the room temperature and the temperature of the water in the battery jar or graduated cylinder. (4) *Why is this latter temperature necessary?* Obtain the room pressure.



**Fig. 29-1** Apparatus for Determining the Volume of Hydrogen Produced by a Given Quantity of Magnesium

## Supplementary Questions:

- (5) *What was the water vapor pressure at the measured water temperature?*
- (6) *What was the pressure exerted by the hydrogen alone?*
- (7) *What volume would the dry hydrogen gas occupy at STP?*
- (8) *What volume of hydrogen gas at STP would have been produced by one mole of magnesium?*
- (9) *Given that one mole of magnesium produces one mole of hydrogen gas, what is the molar volume of hydrogen gas at STP determined in this experiment?*
- (10) *What is the percent difference between your answer and the accepted value for the molar volume of hydrogen gas at STP?*

# THE MOLAR MASS OF A VOLATILE LIQUID

## Purpose:

To determine the molar mass of a volatile liquid.

## Introduction:

The mass of one mole of a volatile liquid is determined by adding an excess of the liquid to a flask of known mass. The flask is heated in a hot water bath of known temperature. The liquid evaporates and vapor fills the flask. Excess vapor is lost to the atmosphere.

When the flask is cooled, the vapor remaining in it condenses. The mass of the condensed liquid is found by determining the mass of the flask. The mass of the liquid equals the mass of the vapor that filled the flask at the higher temperature. The volume that this mass of vapor would occupy at STP can be calculated. Since one mole of an ideal gas occupies  $22.4\text{ l}$  at STP, the mass of one mole (molar mass) of the volatile liquid can be found.

## Apparatus:

125 cm <sup>3</sup> Erlenmeyer flask	wire gauze
10 cm <sup>3</sup> graduated cylinder	burner
pin	filter paper
buret clamp	250 cm <sup>3</sup> graduated cylinder
ring stand	600 cm <sup>3</sup> beaker
iron ring	

## Materials:

aluminum foil  
volatile liquid  
boiling chips

## Experiment:

- Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

**DATA TABLE**

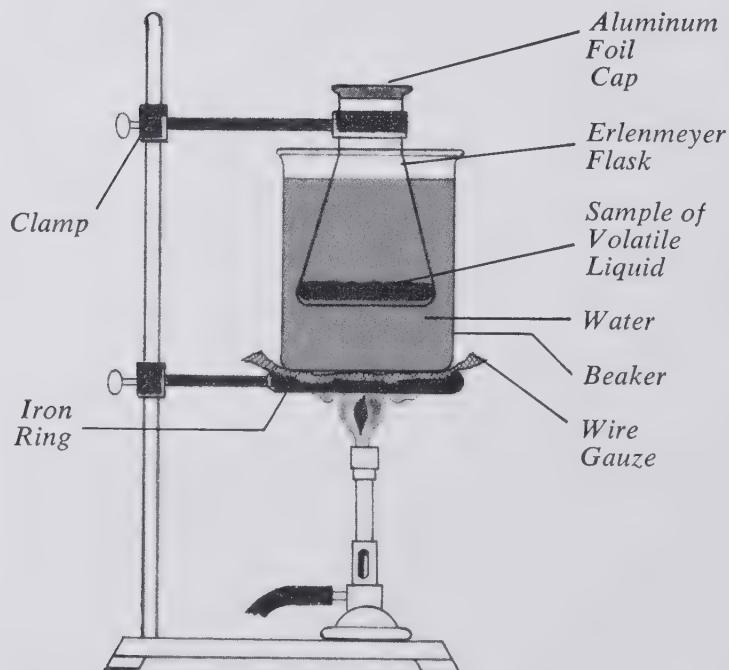
Mass of flask and foil together	_____ g
Mass of flask, foil, and condensed liquid	_____ g
Mass of condensed liquid = mass of vapor	_____ g
Temperature	_____ °C
Barometric pressure (obtained from teacher)	_____ kPa
Volume of flask	_____ l

**B.** Determine the mass of a 5 cm square of aluminum foil and a 125 cm<sup>3</sup> Erlenmeyer flask together.

**C.** Pour 3 cm<sup>3</sup> of an unknown volatile liquid into the flask. Prepare a cap for the flask by placing the aluminum foil on the mouth of the flask and folding it tightly about the mouth of the flask. Use a pin to make a *tiny* hole in the center of the foil.

**D.** Clamp the flask to a ring stand and suspend it in a 600 cm<sup>3</sup> beaker. Tilt the flask slightly so that you can see the liquid. Add a few boiling chips to the beaker and pour water into the beaker so that the flask is surrounded with the water (Fig. 30-1).

**Fig. 30-1** Apparatus for Evaporating a Volatile Liquid



E. Heat the beaker until the last traces of liquid have gone from the flask. (It is not necessary to heat the water to a vigorous boil.) Measure the temperature of the water bath. When all of the liquid has evaporated from the flask, remove the flask from the beaker. Allow the flask to cool to room temperature.

F. Wipe the outside of the flask, including the cap, with filter paper to dry it. Be sure to remove any drops of water which may be trapped between the foil and the glass. Determine the total mass of the flask, the cap, and the condensed liquid.

G. Remove the cap from the flask and fill the flask to the top with tap water. Measure the volume of the flask by pouring the water into a  $250 \text{ cm}^3$  graduated cylinder.

## Questions :

- (1) *What would be the volume of the vapor at STP?*
- (2) *What is the calculated molar mass of your unknown?*
- (3) *What is the molecular mass of your unknown?*
- (4) *If you had made a rather large hole in the foil cap, what effect would this have had on your calculated molar mass?*

# TWO HYDROCARBONS— METHANE AND ETHYNE

## Purpose:

To prepare methane and ethyne and examine some of their properties.

## Introduction:

Methane and ethyne are both hydrocarbons. Methane is a typical member of the alkane series of hydrocarbons, and ethyne is a typical member of the alkyne series.

## Apparatus:

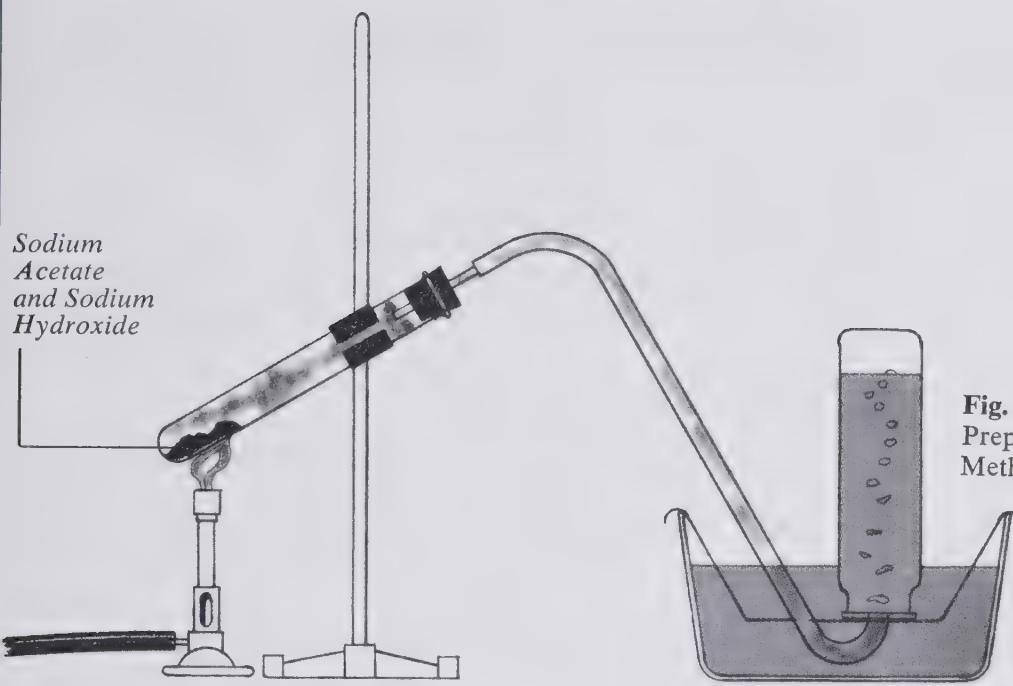
200 mm test tubes (2)	rubber tubing
buret clamp	pneumatic trough
ring stand	wide-mouthed bottles (2)
burner	rubber stopper for bottle
one-hole stopper for test tube	rubber stopper for test tube
short piece of glass tubing	spatula

## Materials:

sodium acetate (anhydrous)	bromine water
sodium hydroxide	calcium carbide
wooden splints	

## Experiment:

- A. Set up a methane generator as shown in Fig. 31-1. Place a mixture of 8 g of anhydrous sodium acetate and 4 g of sodium hydroxide in a test tube. Spread the mixture evenly in the test tube so that there is an air space above the mixture. Place two inverted bottles full of water and one inverted test tube full of water in the pneumatic trough. Insert the delivery tube into the mouth of one of the bottles.
- B. Heat the contents of the test tube evenly. Collect half a bottle of gas and discard it. (*1. Why is this gas discarded?*) Collect one bottle and one test tube of methane gas. Remove the end of the delivery tube from the water before



**Fig. 31-1** Apparatus for the Preparation and Collection of Methane

removing the flame from the generator. (2) *Why must the delivery tube be removed from the water before heating is stopped?* (3) *What physical properties of methane do you observe?*

- C.** Thrust a lighted splint into the bottle of methane. (4) *What do you observe?*
- D.** To the test tube of methane add 2 cm<sup>3</sup> of bromine water. Stopper the test tube and shake it vigorously. (5) *What do you observe?* Remove the stopper and exhale across the mouth of the test tube. (6) *What do you observe?*
- E.** To generate ethyne, first place one inverted bottle and one inverted test tube, both full of water, in the pneumatic trough. Then, using a spatula, drop a few pieces of calcium carbide into the trough. Move the bottle over the ethyne bubbles being evolved. Collect a full bottle of ethyne, and then collect a full test tube of ethyne. (7) *What physical properties of ethyne do you observe?*
- F.** In the fume hood, thrust a lighted splint into the bottle of ethyne. (8) *What do you observe?* (9) *Why must this operation be performed in the fume hood?*

**G.** To the test tube of ethyne add 2 cm<sup>3</sup> of bromine water. Stopper the test tube and shake it vigorously. (10) *What do you observe?* Remove the stopper and exhale across the mouth of the test tube. (11) *What do you observe?*

### Supplementary Questions:

- (12) *How do the behaviors of methane and ethyne toward a lighted splint compare? How do they differ?*
- (13) *How do the reactions of methane and of ethyne with bromine water compare? How do they differ?*
- (14) *Hydrogen bromide is so soluble in water that it causes the water vapor in your breath to condense and form visible droplets. Is hydrogen bromide produced during this experiment? If it is produced, where is it produced, and what does this indicate about the reaction taking place?*
- (15) *For what purpose is methane used most? What well-known commercial substance consists chiefly of methane?*
- (16) *What is the common commercial name of ethyne? For what purpose is ethyne used most?*

# PREPARATION OF ETHANOL BY FERMENTATION

32

## Purpose :

To prepare ethanol and identify some of its properties.

## Introduction :

When grain, tubers, and fruits ferment, the carbohydrates in them are converted by a series of reactions into ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$ . Various enzymes contained in yeast speed up the reactions involved.

Prior to this experiment your teacher mixed a cake of yeast with sugar and warm water. This mixture was added to a larger bottle containing  $2500 \text{ cm}^3$  of warm water and  $500 \text{ cm}^3$  of molasses. This mixture has been quietly fermenting for several days.

## Apparatus :

250  $\text{cm}^3$  distillation flask or Florence flask  
buret clamp  
condenser clamp  
thermometer  
bent glass tube ( $60^\circ$ )  
two-hole rubber stopper for flask  
wire gauze  
iron ring  
ring stands (2)  
burner  
condenser  
one-hole rubber stopper for condenser  
pieces of rubber tubing (2)  
 $10 \text{ cm}^3$  graduated cylinder  
evaporating dishes (3)  
large watch glass

## Materials :

molasses and yeast mixture  
boiling chips  
wooden splints

## Experiment:

A. In a  $250\text{ cm}^3$  flask place  $150\text{ cm}^3$  of the fermented liquid and a boiling chip. Arrange the apparatus as shown in Fig. 32-1. Run cold water through the condenser as shown. Heat the solution with a burner, and collect the distillate in a  $10\text{ cm}^3$  graduated cylinder. (1) *At what temperature does the first drop of distillate fall into the receiver?*

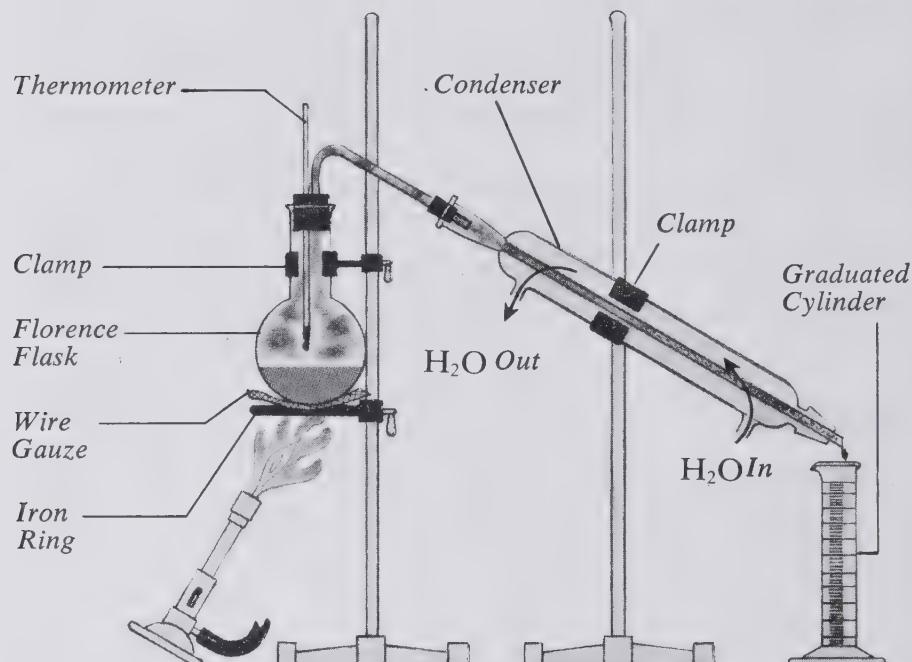


Fig. 32-1 Apparatus for Distilling Ethanol

B. Collect three  $10\text{ cm}^3$  portions of distillate and place these portions in evaporating dishes. (2) *What are the temperatures at the beginning and end of each  $10\text{ cm}^3$  collection?* (3) *What is the odor of each portion?* Bring a burning splint to the surface of each portion. (4) *Does any portion ignite?* If so, extinguish the flame immediately by covering the dish with a watch glass.

C. Empty the distillation flask. Place in it the three combined portions of distillate and a fresh boiling chip. Collect a  $10\text{ cm}^3$  portion of distillate. (5) *What are the temperatures at the beginning and at the end of this distillation?* (6) *What is the odor of this portion of distillate?* (7) *Does this portion ignite when tested with a burning splint as described in part B?* (8) *What are the properties of ethanol that you were able to observe?*

## Supplementary Questions:

- (9) If any portion of distillate ignited, what was the boiling temperature range of the portion which burned?
- (10) What is the chemical equation for the preparation of ethanol from sucrose ( $C_{12}H_{22}O_{11}$ )?
- (11) What is the chemical equation for the combustion of ethanol?
- (12) What are the most important industrial uses of ethanol?

# PERCENTAGE OF ACETIC ACID IN VINEGAR

## Purpose:

To determine the percentage by mass of acetic acid in vinegar.

## Introduction:

Acetic acid is produced by the bacterial oxidation of ethanol. Apple cider is often the source of this ethanol. The process stops when the concentration of acetic acid becomes large enough to prevent the bacteria from continuing the oxidation of ethanol. The resulting solution of acetic acid is called vinegar.

In this experiment you will titrate the acetic acid in a known mass of vinegar with sodium hydroxide solution of a known concentration. The sodium hydroxide will neutralize the acetic acid. Phenolphthalein is added to indicate the point at which the acetic acid has been completely neutralized by the sodium hydroxide.

A knowledge of the concentration (moles per litre) and the volume (litres) of the sodium hydroxide required to neutralize the acetic acid enables us to calculate the number of moles of sodium hydroxide used. Since acetic acid has one ionizable hydrogen atom per molecule, one mole of sodium hydroxide will neutralize one mole of acetic acid. Hence, the number of moles of acetic acid present in the vinegar can be obtained and used to calculate the mass of acetic acid in the known mass of vinegar.

## Apparatus:

150 cm<sup>3</sup> beaker  
50 cm<sup>3</sup> buret  
buret clamp

ring stand  
250 cm<sup>3</sup> Erlenmeyer flasks (2)  
50 cm<sup>3</sup> graduated cylinder

## Materials:

vinegar  
1.00 M sodium hydroxide  
phenolphthalein solution

## Experiment:

A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete

the remainder of the data table as soon as you have enough data to do so.

DATA TABLE	#1	#2
Mass of flask and vinegar	_____ g	_____ g
Mass of empty flask	_____ g	_____ g
Mass of vinegar	_____ g	_____ g
Final reading of buret	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Initial reading of buret	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Volume of NaOH used	_____ l	_____ l
Moles of NaOH used	_____ mol	_____ mol
Moles of acetic acid in vinegar	_____ mol	_____ mol
Mass of acetic acid in vinegar	_____ g	_____ g
Percentage of acetic acid in vinegar	_____ %	_____ %

**B.** Label two Erlenmeyer flasks 1 and 2. Determine the mass of each flask. Rinse a graduated cylinder with 5 cm<sup>3</sup> of vinegar, then measure 20 cm<sup>3</sup> of vinegar into each flask. Determine the total mass of each flask containing the vinegar. Add 1 drop of phenolphthalein solution to each flask. (1) *What is the color of phenolphthalein in vinegar?*

**C.** Obtain about 100 cm<sup>3</sup> of 1.00 M sodium hydroxide in a 150 cm<sup>3</sup> beaker. Rinse the buret with about 10 cm<sup>3</sup> of this solution and let the liquid drain through the buret tip. Repeat this procedure once more. Refill the buret to a location above the zero mark and fasten it to a ring stand with a buret clamp. (2) *Why must the buret be rinsed with sodium hydroxide solution before it is filled?* Let some of the sodium hydroxide solution run rapidly from the buret to expel all air bubbles from the tip and to bring the level of the solution down to the calibrated region of the buret. Hold a piece of white filter paper behind the meniscus and read the initial volume of the liquid at the level of the bottom of the meniscus (see Fig. 2-1). The meniscus is the curved surface of the liquid.

**D.** Place one of the Erlenmeyer flasks under the tip of the buret. A piece of white filter paper placed under the flask will make it easier to see the color changes. With continuous swirling of the flask to ensure thorough mixing, run in the sodium hydroxide solution from the buret until the color which

forms as the sodium hydroxide hits the solution does not disappear quickly. (3) *What color change do you observe?* Then add the sodium hydroxide solution more slowly, finally adding it drop by drop until one drop is sufficient to cause a permanent color change. This color change indicates that enough sodium hydroxide has been added to neutralize the acetic acid in the vinegar. The volume of sodium hydroxide in cubic centimetres must be converted to litres in order to complete the calculations.

E. Repeat the titration with the second flask.

# ESTERS

## Purpose:

To study a method for making esters, and to study some of their properties.

## Apparatus:

250 cm <sup>3</sup> beaker	wire gauze
burner	medicine dropper
ring stand	evaporating dish
iron ring	150 mm test tubes (4)

## Materials:

ethyl alcohol	salicylic acid
acetic acid	methyl alcohol
amyl alcohol	sulfuric acid

## Experiment:

- A. Heat 200 cm<sup>3</sup> of water in a 250 cm<sup>3</sup> beaker to the boiling point. While the water is heating, label four clean, dry test tubes A, B, C, D.
- B. In test tubes A and B place 10 drops of ethyl alcohol and 10 drops of acetic acid. In test tube C place 10 drops of amyl alcohol and 10 drops of acetic acid. In test tube D place enough salicylic acid to fill it 1 cm deep. Add 3 cm<sup>3</sup> methyl alcohol. Place three drops of concentrated sulfuric acid in tubes B and C, and five drops of sulfuric acid in tube D. Mix the contents of all four tubes well. (1) *Describe as closely as possible the odor of the contents of each of the tubes.*
- C. Place all four tubes in the boiling water for 5 min. Then pour the contents of tube A into an evaporating dish half-full of cold water. (2) *Describe the odor of the contents of the evaporating dish.*
- D. Rinse the evaporating dish and pour the contents of test tube B into the evaporating dish half-full of cold water. (3) *Identify the odor of the contents of the dish.* (4) *What substance produces the odor?* (5) *Do the odors in tubes A and B differ from each other?* (6) *If so, in what way do they*

differ? (7) What can you say about the effect of the sulfuric acid?

- E. Again, rinse the evaporating dish and pour the contents of tube C into an evaporating dish half-full of cold water. (8) Identify the odor. (9) What substance produces the odor?
- F. Finally, pour the contents of tube D into an evaporating dish half-full of cold water. (10) Identify the odor. (11) What substance produces the odor?

### Supplementary Questions:

- (12) What are the chemical equations for the reactions that occurred in each case? Was there any case in which no reaction occurred? If so, which case was it?
- (13) What substance, besides a carboxylic acid and an alcohol, is required to prepare an ester? How do you know?
- (14) How would you prepare an ester from acetic acid and n-butyl alcohol? Give an equation for the reaction.
- (15) On the basis of this experiment, suggest a likely commercial use for esters.

## POLYMERS

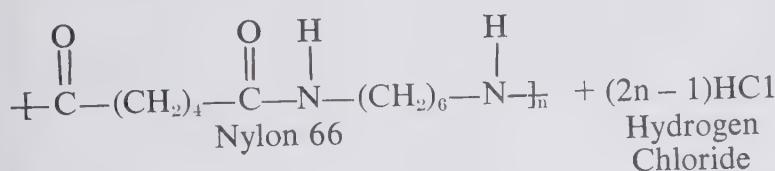
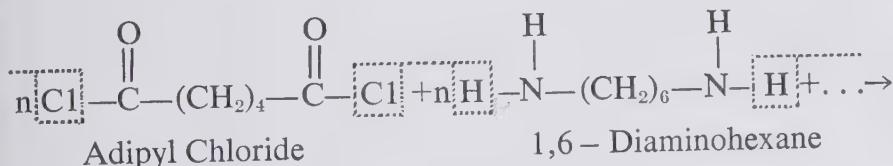
35

## Purpose:

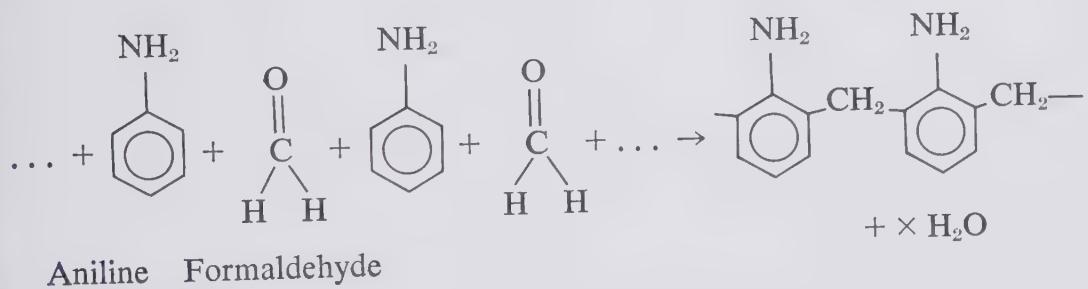
To prepare two polymers.

## Introduction:

The reaction of a dicarboxylic acid chloride with a diamine forms a polyamide molecule. Nylon 66 is made from adipyl chloride and 1,6-diaminohexane (hexamethylenediamine). Each of the two monomers used to make nylon 66 has six carbon atoms:



You will prepare nylon and you will also prepare another type of polymer by using formaldehyde and aniline:



## Apparatus:

10 cm<sup>3</sup> graduated cylinder  
150 mm test tubes (2)  
100 cm<sup>3</sup> beakers (3)  
forceps or bent copper wire

tin can  
stirring rod  
medicine dropper  
 $250 \text{ cm}^3$  beaker

## Materials:

cyclohexane  
5% adipyl chloride solution  
5% 1,6-diaminohexane solution  
5 M sodium hydroxide  
0.2 M acetic acid  
40% formaldehyde solution  
saturated aniline hydrochloride solution

## Experiment:

- A. Place 2 cm<sup>3</sup> of cyclohexane in a 150 mm test tube. Add 5 cm<sup>3</sup> of water to the same test tube. (1) *Are the two liquids miscible?* (2) *If the liquids are not miscible, which liquid is in the bottom layer?* (3) *How do you know?*
- B. Place 10 cm<sup>3</sup> of a 5% solution of adipyl chloride dissolved in cyclohexane in a 100 cm<sup>3</sup> beaker. Place 10 cm<sup>3</sup> of a 5% solution of 1,6-diaminohexane dissolved in water in a second 100 cm<sup>3</sup> beaker and add 10 drops of 5 M sodium hydroxide.
- C. Gently pour the adipyl chloride solution down the wall of the beaker containing the diamine solution. (4) *What do you observe?* (5) *If there is any evidence of polymer formation, what is the evidence?*
- D. Use a pair of forceps or a bent copper wire to hook the polymer where it has formed. Slowly draw the forceps or copper wire from the beaker so that the polymer is pulled out of the liquid. Wash the nylon polymer in a beaker containing about 200 cm<sup>3</sup> of 0.2 M acetic acid. (6) *Why should the nylon be washed in a dilute acid solution?* (7) *What properties of the nylon polymer do you observe?*
- E. Place 10 cm<sup>3</sup> of 40% formaldehyde solution and 10 cm<sup>3</sup> of a saturated aqueous solution of aniline hydrochloride in separate test tubes. Simultaneously pour the solutions into a small beaker or preferably a tin can and stir the mixture. As you stir touch the outside wall of the container lightly with your fingers. (8) *What do you observe?* Continue to stir the mixture. (9) *If there is any evidence of polymer formation, what is the evidence?* (10) *What properties of this polymer do you observe?*

## **Supplementary Questions:**

- (11) *What type of polymer (condensation or addition) is being formed in part C? In part E?*
- (12) *What are some uses for nylon with which you are familiar?*
- (13) *What are the names of and uses for other polymers with which you are familiar?*

# THE PREPARATION AND PROPERTIES OF A SOAP

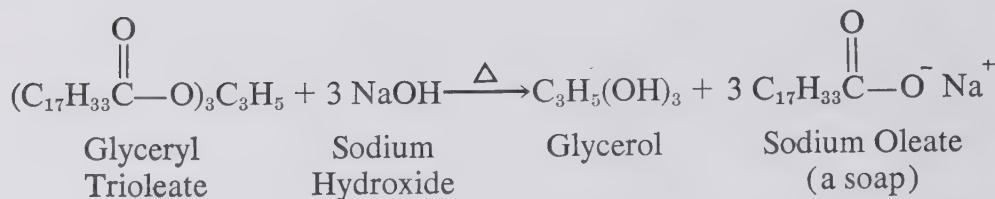
## Purpose:

To prepare a soap and investigate some of its properties.

## Introduction:

The reactions between alcohols and acids produce esters. Fats and oils are two types of esters. Fats are mixtures of esters made from glycerol and carboxylic acids such as stearic, oleic, and palmitic acid. Oils, such as olive oil or cottonseed oil, contain more of the glyceryl esters of oleic acid. Solid fats contain more of the glyceryl esters of stearic and palmitic acid.

Common soap is made by reacting a fat or an oil with a concentrated solution of sodium hydroxide. The product of the reaction (*soap*) is a sodium salt of the acid from which the fat or oil was originally made. The reaction is called *saponification*:



In this experiment cottonseed oil and sodium hydroxide are the reactants. The products are soap and glycerol. Ethyl alcohol is used to dissolve both of the reactants and thus speed up the reaction.

## Apparatus:

10 cm <sup>3</sup> graduated cylinder	ring stand
50 cm <sup>3</sup> graduated cylinder	watch glass
evaporating dish	burner
stirring rod	150 mm test tubes (2)
wire gauze	filter paper
iron ring	funnel

## Materials:

cottonseed oil or olive oil  
ethanol  
10 M sodium hydroxide  
saturated sodium chloride solution  
kerosene  
saturated calcium sulfate solution  
lampblack  
phenolphthalein

## Experiment:

- A. Place 4 cm<sup>3</sup> of cottonseed oil or olive oil and 5 cm<sup>3</sup> of ethanol in an evaporating dish. Add, with stirring, 3 cm<sup>3</sup> of sodium hydroxide solution. Place the evaporating dish on a wire gauze on a ring stand and cover the dish with a watch glass. Heat gently, keeping the flame away from the top of the evaporating dish to prevent the alcohol from catching fire as it leaves the evaporating dish. Continue to heat until the odor of the alcohol disappears. Allow the contents of the evaporating dish to cool. *(1) What do you observe in the evaporating dish?*
- B. Add 20 cm<sup>3</sup> of saturated sodium chloride solution to the contents of the evaporating dish. *(2) What do you observe?* After stirring the mixture well, filter off the solid product. *(3) What physical properties of the soap do you observe?*
- C. Place 1 cm<sup>3</sup> of kerosene and 10 cm<sup>3</sup> of water in a test tube and shake it vigorously. *(4) What do you observe?* Transfer a small amount of your soap to the same test tube and again shake vigorously. *(5) What do you observe?* If there is no change, add more soap and shake the test tube again. *(6) What effect does the addition of soap have on the water-kerosene mixture?*
- D. Place one gram of lampblack in each of two test tubes. Add 5 cm<sup>3</sup> of water to each test tube. To one of the test tubes add a small portion of your soap. Shake both test tubes vigorously. Empty both test tubes, rinse them with water, and observe whether they are free of lampblack. *(7) What effect does the addition of soap have on the water-lampblack mixture?*

- E. Dissolve a small amount of your soap in 5 cm<sup>3</sup> of warm distilled water. Add several drops of calcium sulfate solution. (8) *What effect does the calcium sulfate have on the soapy water?*
- F. Dissolve a small piece of your soap in 5 cm<sup>3</sup> of ethanol. Add two drops of phenolphthalein. (9) *What do you observe?* (10) *What do you learn by adding the phenolphthalein?*

### **Supplementary Questions:**

- (11) *We often hear that the home-made lye soap of the pioneers was very harsh to the skin. What could have been the reason for this?*
- (12) *Do you think the soap you prepared is likely to be harsh? Why?*

# CARBOHYDRATES

37

## Purpose:

To study the reactions of different classes of carbohydrates with specific reagents, and to use the results of these reactions to identify an unknown carbohydrate.

## Introduction:

Carbohydrates react with many different reagents, usually to give colored products. The specific reactants are named after the chemists who discovered them, and in many cases the exact structure of the products is still not known. The value of the tests lies in the fact that different results are obtained depending on the nature of the carbohydrate, that is, on whether it is a monosaccharide or starch, an aldose or a ketose, a pentose or a hexose, and so on.

You will be given an aqueous solution of an unknown carbohydrate which is either ribose, glucose, fructose, or starch. Once you have determined the effects of each reagent on known compounds, you can use this knowledge to identify your unknown.

When running a test for the first time it is desirable to run a "control" or "blank" which contains no carbohydrate. This shows what the reagent alone looks like and gives a basis for comparison when trying to decide whether the color or some other property of a solution has changed. Also, when attempting to identify an unknown, it is a desirable practice to run known solutions (as well as blanks) along with the unknown. Exact times required for reaction, the color produced, and other variables may depend on the age of the reagent, concentration of solution, and other factors.

## Apparatus:

medicine dropper	iron ring
150 mm test tubes (12)	ring stand
10 cm <sup>3</sup> graduated cylinder	burner
400 cm <sup>3</sup> beaker	evaporating dish
wire gauze	filter paper

## Materials:

1% ribose solution  
1% glucose solution  
1% fructose solution  
1% starch solution  
1% unknown carbohydrate solution  
Bial's reagent  
Seliwanoff's reagent  
Molisch reagent  
18 M sulfuric acid  
0.05% iodine solution

## Experiment:

A. Prepare a large data table as shown. Record all experimental results in the data table as soon as you obtain them.

DATA TABLE	TEST			
	BIAL	SELIWANOFF	MOLISCH	IODINE
Ribose				
Glucose				
Fructose				
Starch				
Unknown				
Blank (water)				

B. *Bial's Test.* For this part you will test solutions of ribose, glucose, an unknown, and a blank. First, place about 200 cm<sup>3</sup> of tap water in a 400 cm<sup>3</sup> beaker and heat it to boiling on the ring stand. While the water is heating, place about 1 cm<sup>3</sup> (20 drops) of each substance to be tested in separate test tubes. Add approximately 2 cm<sup>3</sup> of Bial's reagent to each test tube. Mix well and place them in the water bath when it reaches the boiling point. Observe any color changes during 15 min of heating. (1) *What is a negative result (obtained from the blank)?* (2) *Which of the two known carbohydrates gives a negative result?* (3) *Which gives a positive result?* (4) *What is a positive result?* (5) *In what way do ribose and glucose differ (i.e., aldose vs. ketose, pentose vs. hexose, monosaccharide vs. polysaccharide, etc.)?* (6) *What class of carbohydrate is Bial's test useful in distinguishing?*

**C. Seliwanoff's Test.** For this part you will use solutions of glucose, fructose, an unknown carbohydrate, and a blank. To 2 cm<sup>3</sup> of Seliwanoff's reagent in separate test tubes add four drops of the solution to be tested. Mix well and place in the boiling water bath. Record the time required for any changes in color or transparency to occur. Leave tubes in the water bath for ten minutes before recording a negative result.  
(7) *What is a negative result?* (8) *Which of the two known carbohydrates gives a negative result?* (9) *Which gives a positive result?* (10) *What is a positive result?* (11) *In what way do glucose and fructose differ?* (12) *What class of carbohydrate is Seliwanoff's test useful in distinguishing?*

**D. Molisch Test.** For this part you will use solutions of ribose, glucose, fructose, starch, an unknown carbohydrate, and a blank. Place 2 cm<sup>3</sup> of each solution to be tested in separate test tubes. To each test tube add 2 drops of the Molisch reagent and mix well. Tilt each test tube in turn and pour carefully, to avoid mixing, approximately 5 cm<sup>3</sup> of concentrated sulfuric acid down the side of the tube. You should obtain two separate layers.  
(13) *Which layer is the sulfuric acid?* (14) *What do you observe at the junction of the two layers (hold a piece of filter paper behind the test tube for better viewing)?* (15) *What is a negative result?* (16) *Which of the known carbohydrates, if any, gives a negative result?* (17) *For what purpose can the Molisch test be used?*

**E. The Iodine Test.** For this part you will use solutions of ribose, starch, an unknown carbohydrate, and a blank. In an evaporating dish or spot plate add a drop of iodine solution to five drops of each separate solution.  
(18) *What is a negative result?* (19) *Which of the two known carbohydrates gives a negative result?* (20) *Which gives a positive result?* (21) *What is a positive result?* (22) *In what way do ribose and starch differ?* (23) *What kind of carbohydrate is the iodine test useful in distinguishing?*

### **Supplementary Questions:**

(24) *Is your unknown carbohydrate a ketose? How do you know?*  
(25) *Is it a pentose or a carbohydrate with more carbon atoms? How do you know?*  
(26) *Is it a monosaccharide or starch? How do you know?*  
(27) *What carbohydrate is present in your unknown solution?*

(28) Suppose your chemistry teacher had prepared solutions of ribose, glucose, and fructose for this experiment and a student had accidentally mixed up the bottles before they could be properly labeled. What is the minimum number of tests you would have had to perform in order to label the three bottles correctly? Describe what tests you would perform and the information you would obtain from each.

# ENZYMES OF DIGESTION— HYDROLYSIS OF STARCH

## Purpose:

To study the ability of amylase to catalyze the hydrolysis of starch.

## Introduction:

The molecules of the food we eat are converted by the process of digestion into molecules that the body can use as sources of energy or as starting materials for the building of body tissues. Many different enzymes are involved in the digestion process.

An enzyme called amylase found in saliva catalyzes the hydrolysis of starch in the food we eat and thus initiates the process of digestion. In this experiment you will investigate the ability of amylase to catalyze the hydrolysis of starch molecules.

Starch is a polymer consisting of many glucose units joined together. The hydrolysis of starch produces progressively smaller fragments, eventually yielding molecules of glucose and maltose (a disaccharide consisting of two glucose units joined together).

## Apparatus:

150 mm test tubes (6)	wire gauze
10 cm <sup>3</sup> graduated cylinder	burner
400 cm <sup>3</sup> beaker	test tube holder
ring stand	medicine dropper
iron ring	filter paper
stirring rod	

## Materials:

starch	maltose solution
iodine reagent	glucose solution
amylase solution	

## Experiment:

A. Place about 1 g of starch in a test tube. Make a suspension by adding 10 cm<sup>3</sup> of water in 2-3 cm<sup>3</sup> portions with stirring after each addition. Heat the mixture in a boiling water bath for 5 min, stirring the starch suspension from time to time.

(1) *What changes do you observe in the starch mixture?* Re-

move the tube from the water bath. Cool the contents to room temperature by holding the tube under cold tap water. Place 3 drops of the solution in a clean test tube and add 2 drops of the iodine reagent. View the result against a piece of white filter paper. (2) *What do you observe?* (3) *How can this result be used as a test for the presence of starch?*

- B.** Note the time. Then add about 1 cm<sup>3</sup> (20 drops) of the amylase solution to the starch mixture and shake to mix. Let the solution sit and proceed to part C.
- C.** Place 3 drops of a solution of maltose in a test tube and add 2 drops of the iodine reagent. View the result against a white background. (4) *What do you observe?* In the same way, test 3 drops of a solution of glucose with a drop of the iodine reagent. (5) *What do you observe?*
- D.** Ten minutes after you have added the amylase solution, retest the starch solution of part B with the iodine reagent (as you did in part A). (6) *What do you observe?* Continue testing the solution with the iodine reagent every 5 min until the end of the period or until no color is generated by the addition of the iodine reagent. (7) *What is the result of each test? Present your answer in the form of a table.* (8) *How do you explain the results of the successive tests?*

### **Supplementary Question:**

- (9) *If you chewed soda crackers rapidly and swallowed them with a dry mouth, would the starch in them still have a chance of being digested?*

## ANALYSIS OF AN ANTACID

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## Purpose:

To determine how many times its own mass in excess stomach acid an antacid tablet will consume.

## Introduction:

We will assume that stomach acid is approximately 0.14 M hydrochloric acid. Excessive secretion of stomach acid causes the acidity of the stomach to become so great that the pH of the stomach contents falls below 3.0. When this happens, heartburn and indigestion may result. An antacid is said to consume excess stomach acid when it maintains the pH at 3.0 even if more acid is secreted into the stomach.

In this experiment, a crushed antacid tablet is added to 100 g of 0.14 M hydrochloric acid which is used to simulate stomach acid. The antacid will neutralize much of the hydrochloric acid but will leave some of it unreacted. Sodium hydroxide solution, having the same concentration and density as the hydrochloric acid, is then added to the mixture. A given mass of the sodium hydroxide solution will neutralize the same mass of hydrochloric acid. The sodium hydroxide neutralizes the hydrochloric acid which the antacid leaves unreacted. The larger the quantity of hydrochloric acid neutralized by the antacid, the smaller the quantity of sodium hydroxide required to neutralize the remaining acid.

The densities of both the hydrochloric acid and the sodium hydroxide solution are  $1.0 \text{ g/cm}^3$ . Thus, a volume of  $100 \text{ cm}^3$  of hydrochloric acid has a mass of  $100 \text{ g}$ , and the volume (in  $\text{cm}^3$ ) of sodium hydroxide solution is numerically equal to the mass (in g) of the sodium hydroxide solution.

$$\text{Mass of acid neutralized by antacid} + \text{Mass of acid neutralized by NaOH} = \text{Total acid (100 g)}$$

$$\therefore \text{Mass of acid neutralized by antacid} = 100 \text{ g} - \text{Mass of acid neutralized by NaOH}$$

then, 
$$\frac{\text{Mass of acid neutralized by antacid}}{\text{Mass of antacid used}} = \text{Number of times its own mass an antacid tablet will neutralize}$$

## Apparatus:

100 cm <sup>3</sup> graduated cylinder	50 cm <sup>3</sup> buret
250 cm <sup>3</sup> Erlenmeyer flasks (2)	100 cm <sup>3</sup> beaker
mortar and pestle	buret clamp
filter paper	ring stand

## Materials:

0.14 M hydrochloric acid
antacid tablet
4% bromophenol blue solution
0.14 M sodium hydroxide

## Experiment:

**A.** Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the data table as soon as you have enough data to do so.

DATA TABLE	1	2
Mass of hydrochloric acid	100 g	100 g
Mass of sodium hydroxide solution used	_____ g	_____ g
Mass of acid neutralized by antacid	_____ g	_____ g
Mass of antacid used	_____ g	_____ g
Number of times its own mass the antacid tablet will neutralize	_____	_____

**B.** Carefully measure 100 cm<sup>3</sup> (100 g) of hydrochloric acid and place this amount in a 250 cm<sup>3</sup> Erlenmeyer flask.

**C.** Obtain an antacid tablet. Several brands will be tested by the students in your class. Crush the antacid tablet using a mortar and pestle. Determine the mass of the crushed tablet and transfer it to the Erlenmeyer flask.

**D.** Dissolve the antacid in the hydrochloric acid. Some of the inert ingredients used in making the tablet will not dissolve, and the solution will be cloudy. However, this is not important in this experiment.

**E.** Add two drops of a 4% solution of bromophenol blue in alcohol to the acid-antacid mixture. (1) *What color of the bromophenol blue indicator shows that excess acid is present?*

**F.** Rinse a buret with sodium hydroxide solution which has the same concentration (0.14 M) as the hydrochloric acid. Fill to about 1 cm<sup>3</sup> above the zero mark, then carefully adjust the level to zero.

**G.** Add sodium hydroxide from the buret to the acid-antacid mixture until the solution turns from its original color through green to blue. The sodium hydroxide should be added until the solution *just* turns blue. (2) *What does this color change indicate?* The volume (in cm<sup>3</sup>) of the sodium hydroxide solution used is numerically equal to the mass (in g) of the sodium hydroxide solution. If time permits, repeat the procedure using a second antacid tablet of the same brand.

### Supplementary Questions:

(3) *What are the desirable properties of a useful antacid?*

(4) *If several brands of antacid were tested by the class, which brand consumed the most excess stomach acid per gram of antacid?*

(5) *If several brands of antacid were tested, which brand was the best buy? That is, which brand consumed the most excess stomach acid per penny of antacid?*

# DETERMINATION OF VITAMIN C—A TWO-PERIOD EXPERIMENT

## Purpose:

To determine the amount of Vitamin C present in a sample of apple juice.

## Introduction:

Vitamin C, or ascorbic acid, is present in many foods. It is a necessary component of the diet, for its absence leads to the disease known as scurvy. It occurs naturally in citrus fruits (such as oranges, lemons, limes, and grapefruit), in green leafy vegetables (such as turnip greens, spinach, and parsley), and in green peppers and strawberries. Vitamin C is frequently added to foods to enhance their nutritive value and to act as a preservative.

Vitamin C reacts with 2,6-dichloroindophenol, a highly colored dye, to form colorless products. When all the Vitamin C has reacted, a slight excess of indophenol causes the solution to change color. This reaction makes it possible to estimate the amount of Vitamin C present in a sample of a food.

This experiment will require two laboratory periods. In the first period you will react a *known* amount of Vitamin C with the indophenol solution supplied and determine how many milligrams of Vitamin C are consumed by one cubic centimetre of the indophenol solution. In the second session you will use the indophenol solution to determine the Vitamin C present in a sample of apple juice.

## Apparatus:

### Part I

150 cm<sup>3</sup> beaker  
50 cm<sup>3</sup> buret  
buret clamp  
ring stand  
100 cm<sup>3</sup> beaker  
filter paper  
125 cm<sup>3</sup> Erlenmeyer  
flasks (2)  
20 cm<sup>3</sup> pipet

### Part II

50 cm<sup>3</sup> buret  
buret clamp  
ring stand  
100 cm<sup>3</sup> beaker  
filter paper  
125 cm<sup>3</sup> Erlenmeyer  
flasks (2)  
10 cm<sup>3</sup> pipet  
10 cm<sup>3</sup> graduated cylinder  
150 cm<sup>3</sup> beaker

## Materials:

### Part I

0.04% indophenol solution  
0.02% Vitamin C solution

### Part II

9 M sulfuric acid  
formaldehyde solution  
0.04% indophenol solution  
sample of apple juice

## Experiment:

*Part I.* Determination of the amount of Vitamin C that reacts with 1 cm<sup>3</sup> of the indophenol solution.

**A.** Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the table as soon as you have enough data to do so.

Data Table	Trial 1	Trial 2	Trial 3
Initial buret reading	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Final buret reading	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Volume of indophenol used	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Average volume used		_____ cm <sup>3</sup>	
Milligrams of Vitamin C used		_____ mg	
Milligrams of Vitamin C that react with 1 cm <sup>3</sup> of indophenol		_____ mg	

**B.** Obtain approximately 100 cm<sup>3</sup> of the indophenol solution. Rinse a 50 cm<sup>3</sup> buret with about 5 cm<sup>3</sup> of this solution and let the liquid drain through the buret tip. Refill the buret to a location above the zero mark. Clamp the buret to a ring stand and let some of the indophenol solution run rapidly from the buret to expel all air bubbles from the tip and to bring the level of the indophenol down to the calibrated region of the buret. Hold a piece of white filter paper behind the meniscus, and read the volume of the liquid in the buret. (Since the solution is so highly colored, you may find it easier to measure from the top rather than the bottom of the meniscus. It does not matter as long as you are consistent and take all your readings in the same manner.)

- C. Into each of two separate  $125\text{ cm}^3$  Erlenmeyer flasks pipet  $20.0\text{ cm}^3$  of the Vitamin C stock solution labeled "Vitamin C-0.2 mg/cm<sup>3</sup>".
- D. Recheck the level of the meniscus of the indophenol solution in the buret. Place one of the Erlenmeyer flasks under the tip of the buret. A piece of white filter paper placed under the flask will make it easier to see the color changes. With continuous swirling of the flask to ensure thorough mixing, run in the indophenol solution slowly from the buret until the pink color, which forms as the indophenol hits the solution, does not disappear quickly. Then add the indophenol more slowly, finally adding it drop by drop until *one drop* is sufficient to cause a pink color that lasts for 10 s.. Read the volume of the solution in the buret.
- E. Refill the buret and repeat the titration with the second flask.
- F. In each case, the difference between the initial and the final reading represents the volume of indophenol used. If the volumes of indophenol used in the two trials differ by more than 5%, you should make another determination using a third  $20.0\text{ cm}^3$  portion of the Vitamin C stock solution. Repeat until two values agree within 5%.
- G. Using the average of the two best values, calculate the number of milligrams of Vitamin C that react with  $1\text{ cm}^3$  of the indophenol solution.

### Questions for Part I:

- (1) *How would your results have been affected if an air bubble were present in the buret tip at the start of the titration but not at the end?*
- (2) *Why does it not matter if you read your volumes from the top of the meniscus instead of the bottom, as long as you are consistent?*

### Experiment:

#### Part II. Determination of Vitamin C in a sample of apple juice.

- A. Prepare a data table as shown. Record all experimental results in the data table as soon as you obtain them. Complete the remainder of the table as soon as you have enough data to do so.

Data Table	Trial 1	Trial 2
Initial buret reading	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Final buret reading	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Volume of indophenol used	_____ cm <sup>3</sup>	_____ cm <sup>3</sup>
Average volume used	_____ cm <sup>3</sup>	
Milligrams of Vitamin C per 10 cm <sup>3</sup> of apple juice		_____ mg
Milligrams of Vitamin C per 100 cm <sup>3</sup> of apple juice		_____ mg

- B. Into each of two Erlenmeyer flasks pipet 10.0 cm<sup>3</sup> of apple juice. To each flask add 1 cm<sup>3</sup> of 9 M sulfuric acid, 3 cm<sup>3</sup> of formaldehyde solution, and 15 cm<sup>3</sup> of distilled water. Swirl well to mix and allow to stand for 8 min. (The formaldehyde and sulfuric acid combine with sulfur compounds which would interfere with the titration.)
- C. Rinse and fill a 50 cm<sup>3</sup> buret with indophenol solution as in Part I. Measure the initial volume of solution in the buret. Titrate the contents of one of the flasks with the indophenol solution, until one drop just causes a pink color that lasts for 10 s. Read the final volume.
- D. Repeat with the second sample.
- E. Using the average of your two titrations and knowing the number of milligrams of Vitamin C that react with 1 cm<sup>3</sup> of indophenol (from Part I), calculate the number of milligrams of Vitamin C in your 10.0 cm<sup>3</sup> sample of apple juice.
- F. Calculate the number of milligrams of Vitamin C in 100 cm<sup>3</sup> of apple juice.

## Questions for Part II:

- (1) *The sulfuric acid and formaldehyde are added to react with any mercaptans, sulfides, and sulfites which may be present in the juice and react with the indophenol. If you had forgotten to add the sulfuric acid and formaldehyde, how would your values for the amount of Vitamin C have been affected?*
- (2) *Could you use this method to determine the Vitamin C present in tomato juice? Explain.*

# DISSOLVED OXYGEN

## Purpose:

To determine the amount of dissolved oxygen in a water sample.

## Introduction:

Oxygen dissolved in water (dissolved oxygen) is as important to aquatic life as atmospheric oxygen is to us. If water contains about  $7 \text{ mg O}_2/\ell$  or more, it will have enough dissolved oxygen to sustain all aquatic life forms. However, if large quantities of the dissolved oxygen are used up by the oxidation of wastes dumped into the water, fish will not survive. Certain varieties of fish will not survive in water which contains  $5 \text{ mg O}_2/\ell$  or less.

In this experiment, you will be given a sample of water to analyze for dissolved oxygen. Some groups will receive water which has been boiled and placed in a sealed container before it has had a chance to cool down. Some groups will receive water which has been exposed to air for several hours. Other groups will receive water samples from various sources. At the end of the laboratory period, the data obtained by the various groups will be shared.

The procedure involves adding several reagents to the water sample, followed by titration with a solution of sodium thiosulfate. It can be shown that the amount of sodium thiosulfate used in the titration is directly proportional to the amount of dissolved oxygen. The reagents are prepared so that the following equation can be used to obtain the dissolved oxygen in  $\text{mg O}_2/\ell$ :

$$\text{mg O}_2/\ell = 0.40 \times \text{volume of thiosulfate (cm}^3\text{)}$$

## Apparatus:

buret	100 $\text{cm}^3$ graduated cylinder
buret clamp	250 $\text{cm}^3$ Erlenmeyer flasks (2)
ring stand	100 $\text{cm}^3$ beaker
wash bottle	

## Materials:

- water sample in 250  $\text{cm}^3$  screw cap bottle
- 2.15 M manganese(II) sulfate
- alkali-iodide reagent
- 18 M sulfuric acid

0.0050 M sodium thiosulfate  
1% starch indicator solution

## Experiment :

- A. Obtain at least 200 cm<sup>3</sup> of water sample from your teacher.
- B. Place your sample bottle in a 1000 cm<sup>3</sup> beaker or other large container under a dispensing buret filled with manganese (II) sulfate solution. Lower the dispensing buret into the sample bottle until the tip of the buret is below the water surface in the bottle. Add 2 cm<sup>3</sup> of manganese(II) sulfate solution. Since the sample bottle was filled with water to begin with, some liquid will overflow down the walls of the sample bottle. Raise the dispensing buret. Cap the sample bottle, and rinse the walls of the bottle with water from a wash bottle. The beaker will catch this wash water. Do not allow any of the reagents used in this experiment to get on your hands.
- C. Add 2 cm<sup>3</sup> of the alkali-iodide reagent from a second dispensing buret using the procedure described in part B.
- D. After adding the alkali-iodide reagent, shake the sample bottle several times. Allow the precipitate to settle for 3 min. Repeat the shaking and settling operation. Shake the bottle a third time. After the third shaking allow the sample bottle to stand for about 8 min. Proceed to part E.
- E. While waiting for the precipitate to settle, rinse and fill a buret with 0.0050 M sodium thiosulfate solution.
- F. Using the procedure outlined in part B, add 2 cm<sup>3</sup> of concentrated sulfuric acid from a third dispensing buret to the water sample bottle. Mix the liquid by inverting the sample bottle. Iodine (the product of the reactions taking place in the bottle) should be distributed evenly throughout the liquid.
- G. Using a graduated cylinder, pour 100 cm<sup>3</sup> of the water sample into a 250 cm<sup>3</sup> Erlenmeyer flask. Titrate the sample with sodium thiosulfate solution until the solution becomes pale yellow in color. Add three or four drops of starch indicator solution. The solution will turn blue. Continue to add

the thiosulfate solution until the liquid turns from blue to colorless. (1) *What volume (cm<sup>3</sup>) of thiosulfate solution did you use?*

**H.** Pour a second 100 cm<sup>3</sup> portion of sample into a clean Erlenmeyer flask and titrate to a colorless end point as before. (2) *What volume (cm<sup>3</sup>) of thiosulfate solution did you use?* (3) *What was the average volume of thiosulfate solution used in the two titrations?* (4) *How much dissolved oxygen (in mg O<sup>2</sup>/ℓ) was in your sample?*

### **Supplementary Questions:**

(5) *After sharing class data, answer the question: how does the amount of dissolved oxygen in recently boiled water compare with the amount of dissolved oxygen in water that has been exposed to air for several hours?*

(6) *What seems to be the effect of water temperature on the amount of dissolved oxygen?*

(7) *Why is the amount of dissolved oxygen considered to be a good measure of the quality of a body of water?*

# DETERGENTS

## Purpose:

To test for the presence of various additives in detergents.

## Introduction:

One of the problems involved in using soaps as cleansing agents is that their effectiveness is impaired by hard water. Ions such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , and  $\text{Fe}^{3+}$  present in hard water react with soaps, removing them from solution and causing the formation of undesirable precipitates which tend to stick to laundry.

Synthetic detergents have the advantage of being equally effective in hard and soft water. Most detergents contain additives designed to improve their properties and enhance their salability in the marketplace.

Carbonates, silicates, and phosphates are added to many detergents. They appear to aid the cleaning process. They are quite basic and probably exert most of their cleansing action in two ways. First, they react with water to produce hydroxide ions which in turn react with grease and oils to give soaps. Second, they react with the metal ions in hard water to form dense granular precipitates which do not stick to the laundry.

Bleaching agents are included in some detergents to remove undesirable colors from cloth. They are mild oxidizing agents which convert molecules responsible for the color to products which have little or no color.

Most modern laundry detergents contain optical brighteners which have a completely different action from bleaching agents. They act by absorbing invisible ultraviolet light (part of normal sunlight) which falls on them and reemitting a portion of this energy as visible bluish-white light. Cloth which contains optical brighteners adsorbed from the detergent thus appears brighter and whiter.

The detergent filtrate used in this experiment is prepared ahead of time. About 5 g of detergent is heated for one hour in an iron crucible. The heating destroys the organic material in the detergent and the detergent ash is boiled in distilled water. This mixture is filtered and the filtrate is used for the tests in this experiment.

## Apparatus:

10 cm<sup>3</sup> graduated cylinder  
Pyrex® evaporating dish  
stirring rod

150 mm test tubes (5)  
test tube holder  
ultraviolet lamp

## Materials:

detergent filtrate  
concentrated ammonia  
solution  
ammonium chloride  
solid detergent  
red food coloring  
boiling chips

3 M nitric acid  
0.1 M silver nitrate  
saturated ammonium molybdate  
solution  
salt  
flour or starch

## Experiment:

- A.** Place 10 cm<sup>3</sup> of filtrate in an evaporating dish and heat the liquid gently until the volume is reduced to about 2 cm<sup>3</sup>. Slowly add 10 cm<sup>3</sup> of concentrated ammonia solution to the hot liquid. CAUTION: Vapors from hot ammonia solution are irritating to the nose and eyes. Add 1 g of solid ammonium chloride to the mixture, and stir to dissolve the crystals. A white precipitate indicates that silicates are present in the detergent. *(1) Is there any evidence for the presence of silicates in the detergent sample? (2) Why is the volume of the filtrate reduced from 10 cm<sup>3</sup> to about 2 cm<sup>3</sup>?*
- B.** Place about 1 cm<sup>3</sup> of solid detergent in a test tube. Add 15 cm<sup>3</sup> of water and one boiling chip to the test tube, and heat the liquid to dissolve the detergent. Cool the hot detergent solution under running water. Add one very small drop of red food coloring to the detergent solution and pour half of the mixture into a second test tube. Add a fresh boiling chip to the second test tube and heat the contents of this test tube to boiling. Compare the contents of the two test tubes. *(3) Is there any evidence that the detergent contains a bleach? (4) If so, what is the evidence?*
- C.** Place 4 cm<sup>3</sup> of the filtrate and 1 cm<sup>3</sup> of 3 M nitric acid in a test tube. Add 5 cm<sup>3</sup> of 0.1 M silver nitrate. The presence of chloride is indicated by a white turbidity which disappears when excess ammonia solution is added and the test tube is shaken. *(5) Is there any evidence for the presence of chloride in the detergent sample?*

D. Place 4 cm<sup>3</sup> of the filtrate and 2 cm<sup>3</sup> of 3 M nitric acid in a test tube. Add 20 drops of saturated ammonium molybdate solution to the mixture and warm the contents of the test tube if necessary. A yellow precipitate indicates that the detergent contains phosphate. (6) *Is there any evidence for the presence of phosphate in the detergent sample?*

E. Place small quantities of detergent powder, salt, and flour or starch into separate test tubes. View the samples under ultraviolet light in a dark room. CAUTION: Do not look directly at the ultraviolet lamp as this may cause eye damage. (7) *Is there any evidence that the detergent contains an optical brightener which the other solids do not contain?* (8) *If so, what is the evidence?*

### Supplementary Questions:

(9) *Why has the presence of phosphates in detergents caused environmental problems?*

(10) *Does the presence of an optical brightener contribute to the cleaning ability of the detergent?*

# PURIFICATION OF WATER

## Purpose:

To study some methods for the purification of water.

## Introduction:

Filtration and distillation are two methods for purifying water that you have already seen in other experiments. Two other common methods are settling and coagulation. If a sediment-laden solution is allowed to stand undisturbed, the solid particles will slowly settle to the bottom of the container. Alternatively, it is possible to add to the water a chemical that causes a precipitate to form. The newly formed precipitate carries suspended solids with it as it settles fairly rapidly to the bottom of the container. In this experiment you will evaluate the relative effectiveness of the four methods in removing various types of impurity from water.

## Apparatus:

- 10 cm<sup>3</sup> graduated cylinder
- 50 cm<sup>3</sup> graduated cylinder
- 200 mm test tubes (4)
- 150 cm<sup>3</sup> beakers (4)
- stirring rod
- spatula
- filter paper
- funnel
- iron ring
- ring stands (2)
- burner
- wire gauze
- 250 cm<sup>3</sup> Florence flask
- buret clamp
- condenser clamp
- two-hole rubber stopper for Florence flask
- thermometer
- bent glass tube
- one-hole rubber stopper for condenser
- condenser
- rubber tubing (2)

## Materials:

0.01 M aluminum sulfate  
0.01 M calcium hydroxide  
muddy water  
potassium permanganate  
barium sulfate  
boiling chips

## Experiment:

A. In a large test tube place  $16 \text{ cm}^3$  of calcium hydroxide solution. Slowly add  $4 \text{ cm}^3$  of aluminum sulfate solution. (1) *What do you observe?* (2) *If calcium sulfate (soluble) is one product of the reaction, what is the other product?* (3) *What is the balanced equation for the reaction?* In each of three large test tubes labeled A, B, and C, place  $10 \text{ cm}^3$  of muddy water. To tube A add  $10 \text{ cm}^3$  tap water and mix well. To tube B add  $8 \text{ cm}^3$  of calcium hydroxide solution and  $2 \text{ cm}^3$  of aluminum sulfate solution and mix well. To tube C add  $5 \text{ cm}^3$  of tap water,  $4 \text{ cm}^3$  of calcium hydroxide solution, and  $1 \text{ cm}^3$  of aluminum sulfate solution and mix well. Observe each mixture immediately when they are combined and at five minute intervals for the next 20 to 25 min. (4) *What do you observe in each tube as minutes pass?* (5) *On the basis of your observations, would you say that settling is a more efficient or a less efficient method of removing suspended solids from water when calcium hydroxide and aluminum sulfate are added?* (6) *Is the settling process more efficient when small amounts or large amounts of precipitating chemicals are added?* Proceed to part B while the suspensions are settling.

B. To  $100 \text{ cm}^3$  of water in a  $150 \text{ cm}^3$  beaker add a few small grains of potassium permanganate and stir. (7) *Is potassium permanganate soluble in water?* (8) *How do you know?*

C. To  $100 \text{ cm}^3$  of water in another  $150 \text{ cm}^3$  beaker add a spatula full of barium sulfate and stir. (9) *Is barium sulfate soluble in water?* (10) *How do you know?*

D. Filter a portion (about  $10 \text{ cm}^3$ ) of the potassium permanganate-water mixture. (11) *Does filtration remove the potassium permanganate?*

*sium permanganate from water? (12) How do you know? Filter a portion (about 10 cm<sup>3</sup>) of the barium sulfate-water mixture. (13) Does filtration remove the barium sulfate from water? (14) How do you know? (15) How do you explain any difference in the effectiveness of filtration in removing these two types of impurity?*

E. Set up a distillation apparatus as instructed by your teacher (or as in Fig. 32-1). Pour the potassium permanganate-water mixture into the distilling flask. Add a boiling chip, heat to boiling, and collect 5 cm<sup>3</sup> of distillate. (16) *Does distillation remove the potassium permanganate from water? (17) How do you know? (18) For what type of mixture should distillation rather than filtration be used as a method of separation? Explain your answer.*

# PURIFICATION BY CRYSTALLIZATION

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## Purpose:

To study crystallization—a method of purifying impure solids.

## Introduction:

Purification by crystallization depends on the solubility differences of the parts of a mixture. Usually both the desired substance and the impurities are soluble in hot solvent but, on cooling, the desired substance precipitates out leaving the impurities behind in solution. Consider, for example, a mixture of benzoic acid and copper sulfate. Their solubilities in 100 cm<sup>3</sup> of water are:

Benzoic acid: 0.27 g at 0°C and 2.2 g at 100°C

Copper sulfate: 31.6 g at 0°C and 203 g at 100°C

Now consider what will happen if 2 g of a mixture containing about 90% benzoic acid and 10% copper sulfate is added to 100 cm<sup>3</sup> of boiling water. Both will dissolve. When the mixture is cooled to room temperature only the benzoic acid will precipitate out. *All* the copper sulfate and *some* of the benzoic acid will remain in solution. The precipitated benzoic acid can be removed by filtration. The separation from copper sulfate will be complete, but not all the benzoic acid will be recovered. Copper ions clinging to the surface of the precipitated benzoic acid can be removed by washing with small amounts of cold water until the washings no longer contain copper ions as shown by a test: the addition of potassium hexacyanoferrate(II) solution to a solution of copper(II) ions produces a red precipitate of copper(II) hexacyanoferrate(II). When the washings no longer give this precipitate, the precipitated benzoic acid will be reasonably pure and may be dried.

## Apparatus:

filter paper	ring stand
150 cm <sup>3</sup> beaker	burner
100 cm <sup>3</sup> graduated cylinder	funnel
watch glass	150 mm test tube
wire gauze	10 cm <sup>3</sup> graduated cylinder
iron ring	

**Materials:**

- benzoic acid-copper sulfate mixture
- 0.1 M potassium hexacyanoferrate(II)

**Experiment:**

- A. Place 2.0 g of the benzoic acid mixture in a 150 cm<sup>3</sup> beaker  
 (1) *What is the appearance of the crude benzoic acid?* Add 100 cm<sup>3</sup> of water, cover with a watch glass, and heat just to boiling. (2) *What do you observe?* Allow the beaker and contents to cool to room temperature. (3) *What do you observe?*
- B. Filter the cool mixture. Wash the residue in the filter paper by pouring 5 cm<sup>3</sup> portions of cold water over it until the wash liquid no longer gives a red precipitate when tested with a few drops of potassium hexacyanoferrate(II) solution. (4) *What is the residue on the filter paper?* (5) *How does its appearance compare with that of the impure benzoic acid with which you started?* (6) *What substance is present in the filtrate?* (7) *How do you know?* (8) *Is crystallization an effective method for purifying a solid substance?*

**Supplementary Questions:**

- (9) *What is the maximum amount of pure dry benzoic acid you could hope to recover by recrystallizing 2.0 g of 90% pure benzoic acid mixture from hot water and cooling the solution to 0°C?*
- (10) *Aspirin or acetylsalicylic acid is structurally related to benzoic acid. How, do you think, is it purified when it is prepared on a commercial scale?*



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L. J. E SLIP

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DUE OCT 16 '90	
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# INTERNATIONAL ATOMIC MASSES

NAME	SYMBOL	ATOMIC NUMBER	ATOMIC MASS	NAME	SYMBOL	ATOMIC NUMBER	ATOMIC MASS*
Actinium	Ac	89	(227)	Molybdenum	Mo	42	95.9
Aluminum	Al	13	27.0	Neodymium	Nd	60	144.2
Americium	Am	95	(243)	Neon	Ne	10	20.2
Antimony	Sb	51	121.8	Neptunium	Np	93	(237)
Argon	Ar	18	39.9	Nickel	Ni	28	58.7
Arsenic	As	33	74.9	Niobium	Nb	41	92.9
Astatine	At	85	(210)	Nitrogen	N	7	14.01
Barium	Ba	56	137.3	Nobelium	No	102	(255)
Berkelium	Bk	97	(247)	Osmium	Os	76	190.2
Beryllium	Be	4	9.01	Oxygen	O	8	16.00
Bismuth	Bi	83	209.0	Palladium	Pd	46	106.4
Boron	B	5	10.8	Phosphorus	P	15	31.0
Bromine	Br	35	79.9	Platinum	Pt	78	195.1
Cadmium	Cd	48	112.4	Plutonium	Pu	94	(244)
Calcium	Ca	20	40.1	Polonium	Po	84	(209)
Californium	Cf	98	(251)	Potassium	K	19	39.1
Carbon	C	6	12.01	Praseodymium	Pr	59	140.9
Cerium	Ce	58	140.1	Promethium	Pm	61	(145)
Cesium	Cs	55	132.9	Protactinium	Pa	91	(231)
Chlorine	Cl	17	35.5	Radium	Ra	88	(226)
Chromium	Cr	24	52.0	Radon	Rn	86	(222)
Cobalt	Co	27	58.9	Rhenium	Re	75	186.2
Copper	Cu	29	63.5	Rhodium	Rh	45	102.9
Curium	Cm	96	(247)	Rubidium	Rb	37	85.5
Dysprosium	Dy	66	162.5	Ruthenium	Ru	44	101.1
Einsteinium	Es	99	(254)	Samarium	Sm	62	150.4
Erbium	Er	68	167.3	Scandium	Sc	21	45.0
Europium	Eu	63	152.0	Selenium	Se	34	79.0
Fermium	Fm	100	(253)	Silicon	Si	14	28.1
Fluorine	F	9	19.0	Silver	Ag	47	107.9
Francium	Fr	87	(223)	Sodium	Na	11	23.0
Gadolinium	Gd	64	157.2	Strontium	Sr	38	87.6
Gallium	Ga	31	69.7	Sulfur	S	16	32.1
Germanium	Ge	32	72.6	Tantalum	Ta	73	180.9
Gold	Au	79	197.0	Technetium	Tc	43	(97)
Hafnium	Hf	72	178.5	Tellurium	Te	52	127.6
Helium	He	2	4.00	Terbium	Tb	65	158.9
Holmium	Ho	67	164.9	Thallium	Tl	81	204.4
Hydrogen	H	1	1.008	Thorium	Th	90	232.0
Indium	In	49	114.8	Thulium	Tm	69	168.9
Iodine	I	53	126.9	Tin	Sn	50	118.7
Iridium	Ir	77	192.2	Titanium	Ti	22	47.9
Iron	Fe	26	55.8	Tungsten (Wolfram)	W	74	183.8
Krypton	Kr	36	83.8	Uranium	U	92	238.0
Lanthanum	La	57	138.9	Vanadium	V	23	50.9
Lawrencium	Lw	103	(256)	Xenon	Xe	54	131.3
Lead	Pb	82	207.2	Ytterbium	Yb	70	173.0
Lithium	Li	3	6.94	Yttrium	Y	39	88.9
Lutetium	Lu	71	175.0	Zinc	Zn	30	65.4
Magnesium	Mg	12	24.3	Zirconium	Zr	40	91.2
Manganese	Mn	25	54.9	—	—	104**	(260)
Mendelevium	Md	101	(257)	—	—	105	(262)
Mercury	Hg	80	200.6				

\*A value given in parentheses denotes the mass of the isotope with the longest known half-life.

\*\*The names and symbols of elements 104 and 105 have not yet been agreed upon internationally.

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